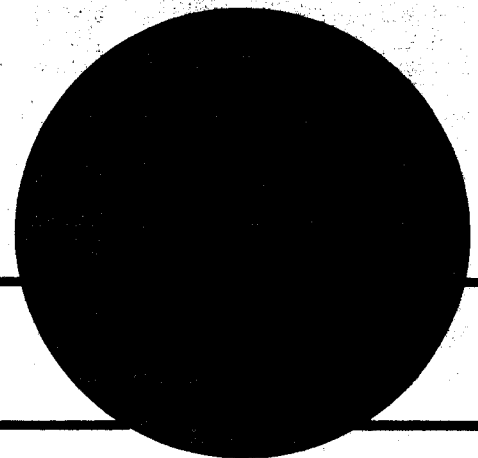


# Materials Processing in Space



Committee on Scientific and Technological Aspects  
of Materials Processing in Space

Space Applications Board

Assembly of Engineering

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*Report of the*  
COMMITTEE ON SCIENTIFIC AND TECHNOLOGICAL  
ASPECTS OF MATERIALS PROCESSING IN SPACE  
*of the*  
SPACE APPLICATIONS BOARD  
ASSEMBLY OF ENGINEERING  
NATIONAL RESEARCH COUNCIL

## MATERIALS PROCESSING IN SPACE

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## NOTICE

The project that is the subject of this report was approved by the Governing Board of the National Research Council, whose members are drawn from the Councils of the National Academy of Sciences, the National Academy of Engineering, and the Institute of Medicine. The members of the committee responsible for the report were chosen for their special competences and with regard for appropriate balance.

This report has been reviewed by a group other than the authors according to procedures approved by a Report Review Committee consisting of members of the National Academy of Sciences, the National Academy of Engineering, and the Institute of Medicine.

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## INTRODUCTION AND SUMMARY

### BACKGROUND

The National Aeronautics and Space Administration (NASA) has long had an interest in materials, particularly those of value in aeronautics and space flight. NASA has carried out extensive studies of materials in its own laboratories and has sponsored materials research in academic and industrial laboratories. Early in the space flight program, interest developed in possible influences the space environment might have on processing materials -- for example, how flow and solidification would occur during fusing of metals in space. From such specific interests emerged a general interest in the possibilities of processing materials in space.

The Space Applications Board (SAB) of the National Research Council (NRC) was established by the National Academy of Engineering in 1972 to provide advice on the uses of space in the national interest. In the course of its work, the SAB in 1974 convened a summer study to consider practical applications of space systems. The study included a panel on materials processing. The report of this panel recommended that the community of materials scientists and engineers be drawn into the planning of NASA's future program on materials processing. As an outgrowth of this recommendation, Dr. James Fletcher, then administrator of NASA, asked the president of the National Academy of Engineering to organize a study, under the direction of the SAB and drawing on the academic and industrial communities, to provide guidance for NASA's program for materials processing in space. Accordingly, in consultation with the Solid State Sciences Committee of the NRC, a plan was developed for a study of materials processing in space, drawing on comments and advice from more than 100 experts in materials science and technology.

To perform the study, the SAB established an ad hoc committee, the Committee on Scientific and Technological Aspects of Materials Processing in Space, whose members (listed on the inside cover of this report) were appointed under the formal procedures of the NRC. The group was interdisciplinary in character, consisting of members drawn from industry, universities, and national laboratories. A few members had previous experience with NASA programs through work on agency-funded research or

through advisory roles; most, however, had little or no previous involvement with NASA. The Solid State Sciences Committee was represented by a liaison member, Shirleigh Silverman.

The overall objective of the study was to provide guidance for the future course of NASA's program of research and development on processing materials in the space environment. The study was also directed toward assessing the scientific and technological underpinnings of the program for materials processing in space and toward providing a clear understanding of the values, if any, to be expected from exploitation of the characteristics of the space environment for processing materials.

The charge to the Committee, as set forth in the NRC plan, was intentionally general. The principal objectives were

- an assessment and evaluation of the scientific and technological significance of what has been learned to date about processing materials in the space environment;

- a judgment of the merit of a program on materials processing in space -- possible benefits, if any; values; advantages and disadvantages; and

- recommendations regarding the nature and scope of NASA's future program of experiments on materials processing in space, as well as on a program of complementary experiments in ground-based facilities or theoretical studies designed to provide a sound scientific basis for the program.

The objectives were viewed by the Committee from the start as being broader than an explicit assessment of processing materials in space. The charge to the Committee was taken to include consideration of the properties and fundamental behavior of materials. The possibility of taking advantage of a low-gravity ("low-g") environment to clarify or broaden the understanding of properties and processes on earth, without any expectation of direct utilization of space for practical purposes, was also seen as an appropriate aim of the study.

At its first meeting in February 1977, the Committee established its method of investigation. The plan provided for the participation of the full Committee in a series of five two-day meetings occurring monthly. Each day's session was devoted to a scientific or technical topic judged important to the objectives of the study. Each session centered on informal presentations by invited guests expert in the topic under review. Questions and discussions further developed the subject. In all, about 60 experts, whose names are included in "Acknowledgments," were heard. On occasion, the Committee invited members of the NASA technical staff and NASA management to participate in its discussions.

A week-long summer workshop in July 1977 concluded the deliberations of the Committee and provided draft materials upon which, in part, this report is based.

## THE SPACE ENVIRONMENT

As used herein, "space environment" refers to the combination of near-zero gravity, high vacuum, electromagnetic fields, and fields of energetic particles that exist within or in the vicinity of a spacecraft in near-earth orbit.

Specialized "drop towers" on earth, aircraft following ballistic trajectories, or sounding rockets can be used to provide near-zero gravity conditions for times ranging from a few seconds to a few minutes. Drop towers suitable for tests of several seconds duration are available at NASA's Marshall Space Flight Center. Ballistic flight of specially modified aircraft can be used for tests of several tens of seconds duration. NASA has developed sounding rockets that can provide near-zero gravity conditions for small payloads for test durations of several minutes.

A major change in accessibility of the space environment will come about 1980 when the Space Shuttle becomes available for materials processing experiments. The Space Shuttle, a reusable spacecraft capable of remaining in near-earth orbit, initially for about seven days, later for periods up to about 28 days, has a 4.6 x 18.3 m (15 x 60 ft) cargo bay. A habitable laboratory, Spacelab, is being developed by the European Space Agency to be carried in the Shuttle cargo bay and will be available about 1981. Experiments may also be placed on pallets outside Spacelab or placed outside the Shuttle in free flight and recovered later.

## THE ROLE OF GRAVITY

This report deals almost entirely with the role of gravity in the science and technology of materials processing in space. The Committee concluded that other aspects of the space environment such as temperature, ambient vacuum, or radiation either had no significant effects on materials or their effects could be duplicated on earth. Although space vehicles such as sounding rockets and the Space Shuttle offer access to several unusual environmental conditions, the aspect of primary interest in the science and technology of materials appeared to the Committee to be the availability of low effective gravitational acceleration for long periods of time. The extent to which a condition of zero gravity can be approached and maintained has much to do with the usefulness of a space facility for materials processing.

In considering the possible role of a low-g environment in materials science and technology, the Committee noted that the influence of gravity in most phenomena is well known. As will be developed later,

gravity is, with rare exceptions, an insignificant force at atomic and molecular levels. It is of no direct consequence in phenomena such as molecular excitation and chemical reaction or on measurable properties of microscopic aggregates of atoms and molecules, with one exception. That exception occurs near phase-transition critical points, where the behavior of matter constitutes a frontier of physical science and where the physical parameters may span the whole of micro, macro, and continuum phenomena. The implications of critical-point phenomena are important to materials science and potentially significant for materials processing on earth. Phenomena near critical points may take minutes to hours to approach steady-state conditions and are legitimate candidates for study in a sustained low-gravity environment.

The behavior of matter at a macroscopic scale -- the scale at which solids, liquids, and gases appear to be continuous -- involves gravity in ways that are well understood. For example, stressing of a solid under its own weight or stressing of a liquid under its own hydrostatic pressure involves familiar mechanical behavior. Similarly, phenomena involving differences in density within fluids are common. Although reducing gravity may have some value for preventing sedimentation, little or nothing new of scientific value is to be learned about these effects through experiments in a low-gravity environment. Neither is there a practical advantage in manipulating these effects that cannot be achieved more simply and less expensively in earth-bound facilities. Other effects of gravity, especially buoyancy-driven convection in fluids, are less well understood, the more so the greater the number of forces, transport processes, heat and mass transfer, chemical reactions and phase changes, solidification, and crystallization.

The magnitude of the gravity vector is not as important as the ratio of the buoyancy force it induces to viscous forces always present in flowing fluids, to inertial forces and surface tension often present, and to electromagnetic forces. There may be several variables in addition to gravity that can be manipulated to reduce convection or avoid its adverse effects. Generally, the more complicated the system, the fewer are the alternatives to reducing gravity when convection cannot be accommodated. Ingenuity of design is an important factor in experiments involving convection.

One can thus identify a set of potential advantages for experiments in a low-gravity environment. The most prominent of these is elimination or reduction of buoyancy-driven convection. In some cases, a continuous reduction is to be expected. In others, a threshold phenomenon will exist such that if a sufficiently low level of gravity is achieved, the practical effects of convection can be eliminated altogether. Another potential advantage lies in the ability to test experimentally the assumptions necessary in theoretical models of inherently complicated systems and to determine which prediction of a theory is correct when the model

gives more than one. Other advantages for experiments in low gravity include reduction of particle settling that otherwise arises from density differences and improvement in isolation of samples by containerless processing.

These considerations depend on the degree to which the gravitational acceleration can be effectively reduced in an orbiting space vehicle. As of this writing, the level of gravity to which experiments will be subjected in the early Space Shuttle flights is uncertain. Wind tunnel data and other evidence indicate that in a low-drag attitude, the resultant acceleration from atmospheric drag will be below  $10^{-6}$  g. Operational considerations will lead to the use of a vehicular attitude that compromises between an orientation aligned with the earth's gravitational gradient and an orientation fixed in inertial space. It appears that this compromise will lead to an effective acceleration of  $10^{-5}$  to  $10^{-4}$  g. Of probably greater importance, however, will be excursions in the gravitational field within the space vehicle caused by movement of the crew and occasional operation of various mechanical systems in the craft. These excursions have not yet been sufficiently well characterized in either size or frequency. They could well be limiting criteria for some experiments, especially those that depend on long intervals at a specified low level of gravity. An improvement in the accuracy of this information will be vital to the development of future plans of would-be investigators both within and outside NASA.

#### SOME GENERAL CONCLUSIONS

The Committee concludes that prospects for using the space environment for research and development on the processing of materials are limited and need to be better defined on a case-by-case basis. The early NASA program for processing materials in space has suffered from some poorly conceived and designed experiments, often done in crude apparatus, from which weak conclusions were drawn and, in some cases, over-publicized. Nevertheless, there is opportunity for meaningful science and technology developed from experiments in space *provided that problems proposed for investigation in space have from the outset a sound base in terrestrial science or technology and that the proposed experiments address scientific or technical problems and are not motivated primarily to take advantage of flight opportunities or capabilities of space facilities*. The Committee has not discovered any examples of economically justifiable processes for *producing* materials in space and recommends that this area of materials technology not be emphasized in NASA's program.

Low gravity appears to offer certain capabilities. These include (but may not be limited to):

to reduce or possibly eliminate buoyancy-driven natural convection;

to reduce density gradients that severely affect critical-point phenomena;

to test experimentally the assumptions necessary in theoretical models of inherently complicated systems;

to reduce the settling of particles; or

to facilitate levitation and isolation of samples.

The Committee believes the question of whether the Space Shuttle and Spacelab can be made useful facilities for significant materials research, development, and processing should be addressed by means of experiments carefully chosen, taking into consideration the information in this report. The facilities should be developed, demonstrated, and, later, if their utility is successfully demonstrated, made available on a reimbursement basis. During development and demonstration (a period of perhaps five or more years), experiments should be funded by NASA. Development of a viable facility in the first phase will require close involvement of potential users and the full cooperation of the materials research community. If the first phase is successful, the Shuttle should assume the character of a national facility comparable to a national laboratory and the cost of its operation should then be paid for by its users. In this, the second phase, the role of NASA should be to maintain and manage the facility, develop it further, and make space and on board equipment available for rent.

#### ORGANIZATION OF THIS REPORT

Subsequent chapters of this report describe the space environment as it affects materials processing, provide some examples of fundamental processes that may be affected by the space environment, and suggest certain technical and management changes to make the program more effective. An appendix provides information on experiments conducted to date.

## THE ENVIRONMENT IN NEAR-EARTH ORBIT AND THE EFFECTS OF GRAVITY ON PHENOMENA IN MATERIALS

### THE ENVIRONMENT IN NEAR-EARTH ORBIT

Several primary environmental parameters have magnitudes and directions in near-earth orbit significantly different from those on earth. Their possible influence on the results of experiments in materials science or materials processing should be considered. Some -- gravity, temperature, pressure, solar and cosmic radiation, and electric and magnetic fields -- are readily identifiable. They influence static and dynamic phenomena in materials and can give rise to forces or stresses on macroscopic bodies.

#### Ambient Atmosphere

Both the pressure and composition of the atmosphere vary with altitude and temperature in the region of space where the Shuttle will usually operate. Total pressures range from  $10^{-6}$  Torr at low altitudes and high temperatures to  $10^{-9}$  Torr at high altitudes and low temperatures. However, pressures at the low end of this range may not be realized because of the presence of gaseous species emitted by the Shuttle and its payload; the effective ambient gas pressure will be anisotropic because the Shuttle will move at a velocity of approximately 8 km/sec relative to the ambient atmosphere. The total density of the atmosphere will vary from  $10^6$  molecules/cm<sup>3</sup> to  $10^9$  molecules/cm<sup>3</sup>, depending on the Shuttle's operating altitude and the exospheric temperature. In most cases, the predominant chemical species is atomic oxygen, with H, He, and N<sub>2</sub> in various proportions making up only about 10 percent of the total. However, the concentrations of the heavier species drop much more rapidly with increasing altitude than do those of H and He, and the concentration of H atoms increases with decreasing temperature. Thus, for example, the densities of O, H, and He are all approximately  $10^6$  molecules/cm<sup>3</sup> at 500 km altitude and 600°K. At normal operating altitudes, the Shuttle will fly in the F<sub>2</sub> layer of the ionosphere and the density of ion-electron pairs will be about  $10^5$  to  $10^6$  per cm<sup>3</sup>, with O<sup>+</sup> as the predominant ion.

## Thermal Environment

The temperature of an object in orbit is determined by radiative heat exchange with the sun, earth, and deep space as well as by the object's internally generated heat. Deep space subtends a little more than half the spherical viewing angle seen from a body in near-earth orbit and acts as a heat sink with unit emissivity and a temperature near 0°K. The earth subtends the rest of the spherical viewing angle and acts as a radiating body with an average temperature of 254°K, producing an average thermal radiation flux of 237 watts/m<sup>2</sup>. However, the exact amount of radiation and its effective temperature vary locally and its spectrum differs in detail from the black body spectrum because of the variable spectral transmittance of the atmosphere.

The solar constant at a distance of 1.0 astronomical unit is 1353 watts/m<sup>2</sup>; the variation around the earth's orbit ranges between +4.6 watts/m<sup>2</sup> at perihelion and -4.4 watts/m<sup>2</sup> at aphelion. Most of the radiant energy is in wavelengths longer than 7300 angstroms. Intensities at wavelengths of less than 2000 angstroms are about 10<sup>-3</sup> watt/m<sup>2</sup> under quiet conditions to 10<sup>-2</sup> watt/m<sup>2</sup> during flares.

## Magnetic and Electric Fields

The earth's magnetic field varies in intensity from 0.52 to 0.22 gauss between altitudes of 200 and 1000 km, and its direction changes from vertical over the magnetic poles to horizontal at the magnetic equator. The ambient electric field in near-earth orbit is approximately -0.5 volt/m, but the electric field in the vicinity of the Space Shuttle will be determined largely by complex vehicle charging effects and interactions with the ambient plasma.

## Radiation Environment

At normal operating altitudes, the Shuttle's radiation environment will be dominated by the omnidirectional fluxes of electrons and protons trapped in the earth's magnetic field. The flux of electrons with energies above 0.5 MeV will vary with altitude and latitude over a range of the order of 10<sup>3</sup> to 10<sup>6</sup>/cm<sup>2</sup>sec. Proton flux will be in the range between 1 and 100/cm<sup>2</sup>sec for energies above 34 MeV.

## THE INFLUENCE OF FACTORS OTHER THAN GRAVITY

The vacuum existing outside a spacecraft in near-earth orbit is easily reproduced or exceeded in facilities on earth. The cost of earth-based facilities, however, goes up rapidly with increases in chamber volume and pumping speed. It has been proposed to develop a large shield, to be deployed outside the Space Shuttle, in the wake of which would be a vacuum of about 10<sup>-13</sup> Torr with higher pumping speed than could be reasonably provided on earth. The Committee has not looked in depth at possible uses for the proposed shield.

The temperatures expected in near-earth orbit do not differ from those achievable on earth. The Committee saw no advantage to be expected from the temperature regime in near-earth orbit.

While solar radiation, particularly the ultraviolet component, affects materials, the Committee concluded that effects of solar radiation should not be expected to be significant in *processing* materials in space. Cosmic ray particles are known to alter static properties and the rate of dynamic processes in solids. Whether these alterations may be significant to materials processing is questionable. The Committee did not explore the matter in depth.

It may be possible in some special cases to capitalize on environmental conditions existing in the immediate vicinity of spacecraft in near-earth orbit. An example is the possibility of using solar radiation for solar furnaces and electrical power supplies. However, the Committee believes that the most useful property of the space environment for materials research is reduced effective gravity.

#### OFFSETTING THE FORCE OF GRAVITY BY FREE FALL

The force of gravity can be offset by the linear acceleration of free fall near the earth's surface. The distance available for free fall near the earth's surface is limited, however, and therefore the time at reduced gravity is short. For example, in a 400-foot drop tower, free-fall time is about five seconds. Increasing the distance to one mile by using a borehole would increase free-fall time to about 18 seconds. For an aircraft flying a parabolic trajectory, practical limitations of speed and altitude restrict the low-gravity period to about 25 seconds. In typical sounding rockets, after the launch thrust is terminated, a free-fall condition can be achieved for several minutes.

An orbiting spacecraft is unique because it can provide a condition of essentially continuous free fall. The force of the earth's gravity on a mass in a spacecraft in a near-earth orbit is only slightly less than at the earth's surface. The mass in orbit, however, experiences a force owing to centripetal acceleration that virtually balances the earth's gravitational force. Both gravity and centripetal acceleration in effect spring from conservative fields and depend on radial distance in almost exactly opposed ways. In orbital flight, their effects almost completely cancel.

In near-earth orbit, however, a spacecraft experiences other forces that determine how closely a free-fall or virtual zero-g condition can be attained. One such force is drag arising from the passage of the spacecraft through the outer fringes of the earth's atmosphere. NASA's Johnson Space Center and Marshall Space Flight Center have provided estimates of acceleration owing to drag and perturbing forces for a

typical Shuttle flight for which the primary mission is materials processing. On such a mission, it is expected that the Space Shuttle will be operated in an attitude stabilized by the earth's gravity gradient, with the plane of the spacecraft wings in the orbital plane and the nose pointing outward from the center of the earth. This orientation provides minimum atmospheric drag for the gravity-gradient stabilized attitude. Acceleration caused by drag is expected to range from  $-3 \times 10^{-6}$  g at 170 km altitude to  $-2 \times 10^{-8}$  g at 560 km.

The oblateness of the earth and irregularities in its mass distribution will cause small perturbations in the Shuttle's orbit. Because the Shuttle will be in a state of free fall, these perturbations will not influence the effective gravity at the Shuttle's center of mass. However, the forces of constraint that make everything in the Shuttle follow the same trajectory as the center of mass will produce accelerations of the order of  $8 \times 10^{-7}$  g per meter of distance from the orbital path of the center of mass and  $6 \times 10^{-6}$  g per meter of separation from the center of mass. These accelerations will be periodic, with the same period as the orbital motion.

Orbital stationkeeping maneuvers will not be necessary on missions devoted to materials processing experiments, so the only thruster firings will be those of the Shuttle's attitude control system. These firings will produce rotation of the spacecraft that will generally be of short duration, unless a uniform rotation must be induced for thermal control. It is not yet clear whether minor torques due to drag and gravity effects will be small enough to permit an entire mission to be flown in the gravity-gradient mode without firing attitude control thrusters, but NASA believes that use of thrusters can be inhibited for periods at least as long as a day. If desired, attitude control maneuvers can be limited to firings of the vernier engines. For equipment in the Spacelab pressurized module, the resulting accelerations are expected to range from  $3.6 \times 10^{-4}$  g to  $4.4 \times 10^{-5}$  g.

Estimates of accelerations from crew motions are based on extrapolation of accelerations measured as Skylab crew members made body movements while attached to the spacecraft by various restraining devices. Correcting for the mass difference between Skylab and the Shuttle configured for a materials processing mission, the net effect of crew motions is expected to be a random "acceleration noise" distributed over a frequency range roughly from 0.1 to 10 Hz with peak amplitude near 1 Hz. With the crew asleep, the amplitude of this background noise can probably be limited to approximately  $3 \times 10^{-5}$  g. When the crew is active, the amplitude is likely to be about ten times as great, even with precautions to maintain a "quiet ship."

Finally, gas venting, fluid dumps, and operation of a flash evaporator (used to reject heat) are expected to produce accelerations of the order of  $10^{-5}$  g. All these events can be scheduled and gas venting and fluid dumps can be inhibited for periods of the order of a day. The frequency with which the flash evaporator must be operated depends

on energy consumption. Since materials processing experiments tend to be large energy users, one would expect that the evaporator would be operated fairly often on materials processing missions.

To plan and assess the probable value of experiments proposed to take advantage of reduced gravity in the Space Shuttle, it will be important to have information about background accelerations more accurate than is currently available. To interpret results of experiments actually flown, it will be essential to have complete records of the acceleration history.

Some of the perturbations in gravity associated with the Shuttle might be avoided by placing experiments outside the Shuttle in free flight. Free-flying experiments, however, will have their own engineering problems such as providing automation, adequate power, temperature stabilization, and other requirements without introducing perturbations in gravity that equal or exceed those in the Shuttle.

#### THE EFFECTS OF GRAVITY ON PHENOMENA IN MATERIALS

When the Committee began its work, the members found that many effects of low gravity on processing materials had been suggested and provided motivation for a number of experiments (see Appendix). The Committee decided at the outset to consider, at a fundamental level, the possible effects of low gravity on materials processing and phenomena. Documentation of prior experiments and testimony by investigators showed that, with only a few exceptions, experiments have not been so analyzed in the past. It became apparent that the direct effects of gravity are few and can be well understood. The Committee concluded that the unique feature of a low-gravity environment in orbit is that it can be maintained for long periods of time.

In examining the influence of gravity, it is convenient to consider phenomena at the molecular level and in continuous matter.

#### PHENOMENA AT THE MOLECULAR LEVEL

The effect of gravity on atomic and molecular energy levels is negligible; in these domains, electric and magnetic forces overwhelmingly dominate. The gravitational field is neglected in all calculations; the justification is that good agreement between experiment and theory is obtained.\* The point is well illustrated by the hydrogen atom, for

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\* Exceptions occur in certain specialized resonance experiments dealing with hyperfine phenomena. See, for example, Werner et al. "Observation of the Phase Shift of a Neutron Due to Precession in a Magnetic Field," Physics Review Letters 35:1053 (1975).

which exquisite agreement between experiment and theory is obtained without taking into account the effects of gravity. Rudimentary estimates indicate that the gravitational potential energy within the hydrogen atom is of the order of  $10^{-31}$  erg, whereas the electric potential energy is about  $10^{-11}$  erg. Although the energy levels of large atoms and molecules are less precisely known than those of hydrogen, for those entities the conclusion also holds that the influence of gravity is negligible. Thus, experiments at low gravity will not show any discernible effect on those local properties of continuous matter that depend on the internal behavior of atoms and molecules.

The measurable properties of assemblies of molecules in liquids and solids experience thermal fluctuations that totally mask the local effect of gravity. Typically, a measured local property such as temperature depends on at least 10,000 molecules. At this count, thermal fluctuations in the property value are 1 percent and the gravitational potential energy difference across the molecular swarm is on the order of  $10^{-25}$  erg, whereas the thermal fluctuation energy at room temperature is a little more than  $10^{-14}$  erg. With the exception of critical phenomena, discussed below, the local equilibrium equations of state, local contact angles, transport coefficients, and intrinsic phase-transformation rate constants are all insensitive to gravitational fields, at least up to the high field strengths that can be attained with ultracentrifugation.

#### AN EXCEPTION: PHENOMENA NEAR CRITICAL POINTS

The exception occurs when phase-transition critical points are approached. In a sense, a critical point may be considered a meeting place between phenomena at the molecular level and phenomena at the continuum level. Near a gas-liquid critical point, for example, compressibility increases, the ordinary hydrostatic action of gravity produces a strong gradient in density, and, in a gravitational field, it becomes impossible to work with homogeneous material. This is also true of certain other kinds of critical points and it seriously complicates study of critical phenomena. As a critical point is approached, the sizes of the molecular swarms that engage in thermal fluctuations grow so large that the gravitational potential energy difference across a swarm becomes significant. Equations of state, transport coefficients, and transformation rate constants then depend on gravity in ways that are not fully understood.

Time becomes an important parameter. The more closely a critical point is approached, the slower are the transport processes by which equilibrium is attained. Equilibration times in centimeter-scale experiments are typically hours. Diffusion coefficients tend toward zero and to achieve compositional equilibrium in multicomponent solutions may take days or longer. None of the dynamic processes in vicinities of critical points is well understood, though they are basically important to materials science.

Thus, of molecular-level phenomena, only those associated with critical points, particularly in fluids, are likely to be altered detectably by a sustained low-gravity environment. Phenomena at the molecular level are more likely to be appreciably altered by the sustained high-gravity environment in a centrifuge. At present it is not clear which approach holds greater promise of definitive results. Both alternatives challenge experimentalists. Experiments on critical phenomena are appropriate to consider for the Space Shuttle and the results would be potentially significant for materials processing on earth.

## PHENOMENA IN CONTINUOUS MATTER

Examining the effects of gravity on solids, liquids, and gases at visible scales, one finds a set of primary effects that are well understood. In the presence of gravity, materials must have a support or container or, if these are to be avoided, some manner of levitation, positioning, and containment. For a supported solid, a primary effect is stressing of material under its own weight. Within a fluid the analogous effect is hydrostatic pressure. In materials processing, the effect most often of consequence is the action of gravity brought about by density differences. This is the phenomenon of buoyancy.

The force of buoyancy on submerged matter is proportional to both the force of gravity and the difference in density between that matter and the surrounding fluid. In the presence of gravity, suspended particles -- gas bubbles, liquid drops, solid pieces -- move downward if they are more dense and upward if they are less dense than the continuous fluid. Sedimentation of microscopic particles and macromolecules occurs in the same way except that Brownian motions caused by molecular-level fluctuations come into play, one consequence even at equilibrium being a vertical concentration gradient of such tiny particles when they differ in density from the fluid.

If mixing and diffusion do not homogenize them first, miscible but non-uniform liquids stratify upward into layers of successively lower density. Immiscible liquids sort out into a similar layering of phases. Once they reach these states, the systems resist stirring; they are stratified stably against convection. On the other hand, delivery of heat or a low-density solute to the bottom or side of a fluid column keeps density low there. The result is usually a rising current of the low density fluid, with a compensatory sinking current of the higher density fluid from above. This type of circulation, which can be intermittent or continuous, steady or varying, extremely weak or quite strong, well ordered or turbulent, is called *buoyancy-driven convection*, *density-gradient-driven convection*, or *natural convection*.

Heating, cooling, latent heat and reaction heat release (or absorption), solute exclusion, and compositional gradients are commonplace in melt processing and vapor processing of materials. In exceptional circumstances, the resulting density variation is strictly vertical,

buoyancy force vanishes, and there is no natural convection. But generally in any gravitational field, buoyancy force, which is well understood, is activated and in turn generates natural convective flow, which is not well understood, and various secondary effects. Of these, the most important are altered heat transfer and solute redistribution. How important these alterations are depends on many factors. In a given process, the secondary effects of gravity are sometimes beneficial, sometimes nil, sometimes deleterious. Often their consequences are unknown because the interactions of buoyancy force with other forces in fluids can be complicated and subtle. Buoyancy-driven convection is thought by some materials scientists to be capricious, untameable, generally deleterious and therefore to be avoided, even by going to a low-gravity environment if possible. The same can be said of buoyancy-caused settling and sedimentation. This viewpoint has provided the motivation for many of the past suggestions for low-gravity experiments -- suggestions that reflect less than complete understanding of the principles of fluid mechanics and convective transport processes. However, as brought out below, there are classes of experiments proposed for low gravity that can survive scrutiny in the light of these and other physical principles.

To summarize, the primary consequences of gravity on materials at the continuum level are

- need for support, container, or levitation;
- stressing of solids;
- hydrostatic pressure in fluids;
- settling of particles freely suspended in fluids;
- sedimentation of colloids and macromolecules; and
- buoyancy force in non-uniform fluids.

Each of these effects, being directly proportional to the magnitude of gravity, can be reduced in proportion to the reduction of gravity. However, to assess the value of low-gravity experimentation for materials science and materials processing, it is necessary to consider the secondary effects that gravity exerts on continuous matter through these primary effects. Most important are the consequences of settling, sedimentation, and buoyancy-driven convection and of reducing these by lowering gravity. These consequences are best understood in terms of the forces with which gravity competes in fluid systems, the results of convection when the forces move fluid, and the possibilities for controlling or eliminating convection. These considerations lead to the identification of a limited set of potential advantages of low gravity, described in the concluding section of this chapter.

#### FORCES THAT VIE WITH GRAVITY IN FLUIDS

Gravity acts in fluids through hydrostatic pressure and, when density varies in certain ways, through buoyancy force. Other forces prominent in moving liquids and gases and in molding fluid interfaces are

pressure and viscous forces, surface tension forces, and inertial forces (accelerations in disguise, in accord with Newton's second law). Other forces sometimes important in materials processing are electric and magnetic forces and forces arising from acoustic and electromagnetic radiation fields. The importance of buoyancy depends not so much on the magnitude of gravity as on the ratio of the buoyancy force to each of the other forces that are active. Indicators of these ratios are the dimensionless numbers used in similarity analysis, such as Rayleigh number, Grashof number, and Bond number.

To alter the effect of buoyancy, an alternative to changing gravity or density difference is to increase the strength of one of the forces with which buoyancy vies. Safest are the viscous forces, a type of internal friction that cannot generate flow but invariably resists it and often redirects it. If other parameters remain constant, viscous forces increase with increasing viscosity and decreasing container size. Other alternatives are surface tension forces and electromagnetic forces. The former are strong only in small systems, however, and the latter often carry large side effects. Nevertheless, in simple cases in which only two or three forces are important and the shape is highly symmetric, the effect of buoyancy can be altered by changing not gravity but another parameter in the key ratio, a parameter such as viscosity, container size, or surface tension. In this way, the effects of sustained low-gravity conditions can be simulated on earth, as in certain neutral buoyancy experiments and small-scale experiments with capillarity and other surface-tension phenomena.

The possibilities for accomplishing such a simulation diminish as the number of forces that are simultaneously important increases and as the symmetry of the system decreases because not all the pertinent ratios can be scaled throughout the system. There are many solidification, combustion, and separation phenomena in which this is the situation. The next chapter provides examples to illustrate some phenomena in continuous matter that warrant consideration for low-gravity experimentation.

## CONVECTION AND ITS EFFECTS IN FLUIDS

Convection is but one cause of flow. A fluid at rest is set in motion when conditions on it violate the requirements of stable mechanical equilibrium. Conditions such as shear blending, mechanical pumping, isothermal vaporization, melting, chemical reaction, or electrical current flow in the presence of an imposed magnetic field lead to what is called *forced* convection.

In a meniscus -- a gas-liquid or liquid-liquid interface -- there are also several mechanisms that can cause convection. One of these is any variation of surface tension or interfacial tension along the meniscus. Because tension depends on temperature and composition, variations of these properties along the meniscus cause tension gradients.

Flow from a region of low tension toward a region of higher tension is referred to as Marangoni flow. The motion that originates in the meniscus is transmitted to the fluids on either side by viscous action. This mechanism is as well understood as buoyancy. The secondary effects Marangoni convection can have are basically the same as those of buoyancy-driven convection. Even when Marangoni convection is present, however, it is usually overwhelmed by buoyancy-driven convection, *except* in small systems, in small zones near menisci in larger systems, or at low gravity. Order-of-magnitude estimates and computer simulation based on the temperature dependence of surface tension indicate that on earth Marangoni flow can be important throughout liquid drops and zones one centimeter or less in diameter. However, not enough data are available to assess the effects of the composition dependence of surface tension, which is known to totally arrest thermally induced Marangoni flow in certain circumstances.

Convection, whatever its cause, has two significant effects. One is the effect of viscous forces and altered pressure on fluid boundaries. As a result, processes at a solidification front can be influenced and the shape of a meniscus altered, even made unstable. The second effect is to alter transport of heat, constituent chemicals, charge, and suspended particles. If convection is absent, these quantities (apart from suspended particles) are transported by diffusion processes alone. Molecular diffusion is relatively slow and the concentration fields it sets up can easily be altered greatly by very weak convection.

Some proposals for materials processing experiments in space are based on the effects of convection on diffusion regimes. Altered concentration distribution may be seen at shutdown of a batch separation process, or at the discharge of a continuous flow separator, as is the case in particle electrophoresis. The altered concentration distribution at a solidification front propagating into a liquid determines the concentration of the freezing solid. Abundant examples exist in crystallization. Under the conditions that often prevail during materials processing, natural convection tends to be dependent on time, sometimes giving rise to lamellar flows, but often giving rise to flows that are chaotic or turbulent. In the extreme, the concentration distributions approach uniformity again and sometimes this effect of strong convection is a boon.

The effects of convection most often important in materials processing are those that occur in what would otherwise be diffusion regimes. The relative importance of convection and diffusion depends on the convective velocity and the concentration or temperature gradient, their degree of alignment, and the diffusivity or conductivity. Similarly, the effects of convection on sedimentation regimes depend on the convective velocity, the particle concentration gradient, the degree of alignment between the two, and the settling rate of the particles. Examples of the effects of these parameters, in the context of materials processing in a low-gravity environment, are presented in the next chapter.

## IMPORTANCE OF CONVECTION IN MATERIALS PROCESSING

The bulk of melt processing and vapor processing of what eventually become solid materials involves substantial heat release at a solidification front or reaction front -- that is, heat release in a thin, more or less curved, sheetlike zone through which the physical or chemical state changes abruptly. Chemical reaction greatly changes composition across the front. Partitioning, solute exclusion, and segregation during phase transformation can also change composition across the front. Besides these effects, there is usually an intrinsic change in density, most dramatically in combustion reactions.

Were it not for this intrinsic density change, it might be possible to deal with material transformation processes totally free from convection, inasmuch as a transformation front could then propagate through a material without inducing relative motion between the material ahead of and behind the front. The density change drives a type of convection that is the manifest companion of solidification, combustion, etc. Convection driven by density differences consists of acceleration of material on one side of the front with respect to that on the other. Ordinarily, this acceleration is locally perpendicular to the front itself and thus reflects and contributes to curvature of the front.

If the heat release, density change, and other processes taking place within the front allowed it to attain and maintain a highly symmetric shape -- i.e., planar, cylindrical, or spherical, depending on whether the front is initiated from a plane, line, or point -- then the resulting density field could share the same symmetry and the accompanying convection could be extremely simple (irrotational with straight streamlines, in fluid mechanics terms). All would be fully describable in elementary mathematical terms. Indeed, the most incisive and useful theories of solidification, combustion, and similar processes are for situations with planar, cylindrical or spherical symmetry. Of these, only planar symmetry fits the earth's gravitational field and admits the possibility of keeping convection horizontal and one-dimensional by means of stable stratification. There are three difficulties, however. First, non-isothermal processes that release heat at the front are of greatest interest; for such processes, stable stratification requires that the front propagate downward. In solidification and vapor deposition, this requirement can be accommodated in principle, but in combustion, material behind the front is fluid, receives heat from the front, and is thereby *unstably* stratified, which can lead to complicated buoyancy-driven convection. The second difficulty is that compositional segregation at the front is as likely to oppose as reinforce the thermally caused density gradient. When the compositional effect opposes, it may actually destabilize an apparently stable density stratification and lead to complicated buoyancy-driven convection. The third difficulty is inescapable: any planar front intersects the walls of the container and at that intersection the front moves relative to the wall, inducing a hydrodynamic boundary layer in the fluid along the wall. A convective edge effect is unavoidable. Thermal and kinetic edge effects are also

scarcely avoidable. They are known to be important in flame propagation but are incompletely assessed in solidification.

Edge effects tend to prevent planar and cylindrical symmetries, but they are absent from spherically symmetric frontal propagation. Thus, to test the basic physical theories of crystallization, burning, and other transformation processes, it is desirable to devise experiments in which spherical symmetry can be achieved. However, the earth's gravitational field is unidirectional on the scale of laboratory experimentation and so the consequences of buoyancy severely limit the possibilities of achieving spherical symmetry. Sometimes, however, these consequences can be avoided in processes that are fast or occur on a small scale, so that the development time of buoyancy-driven convection is long or the flow strength is small. When the desired symmetry cannot be attained by any practicable means of reducing buoyancy or its consequences at one g or in short free-fall experiments, it is reasonable to consider going to a sustained low-gravity environment in order to test experimentally the assumptions inherent in the basic physical theories.

In reality, the processes within and around a transformation front often *do not* allow it to assume a highly symmetric shape. Rather, non-uniformities along the front are inherent and they tessellate or jumble it, steadily in some cases, periodically in others, chaotically in yet others. Generally, the non-uniformities depend on the rate of frontal advance; not uncommonly they first appear when the rate exceeds a critical threshold signaling an instability. In every case, the result is density gradients that vary in direction in the fluid so that stable stratification in the earth's gravity is impossible. Buoyancy-driven convection is generated, although it may be weak; other forces can reduce but not eliminate it totally. Its magnitude and its consequences depend on time scales and length scales influenced or controlled by forces other than gravity.

To test theories of frontal instability and non-uniformity, it is desirable to grow crystals at high ratios of temperature gradient to growth velocity. Yet at reasonable growth velocities, the needed temperature gradients are a driving force for buoyancy-driven convection that leads to either non-uniform growth velocities or non-planar interfaces. A low-gravity environment will allow low growth rates in the presence of large temperature gradients without convective disturbances at the solidification front. Likewise, to probe, characterize, and test mathematical simulations of typically complex solidification and combustion processes, it is desirable to change the magnitude of natural convection. However, reducing buoyancy-driven convection can bare other types of convection it ordinarily masks, for example, surface-tension-driven convection near gas-liquid and liquid-liquid menisci.

Materials processing can also involve particles suspended in fluid, as in well-stirred melt solidification, in fuel-spray combustion, and in certain methods of biological cell separation (e.g., electrophoresis).

There are many possibilities, such as the use of gels or density-gradient columns, for reducing settling or sedimentation, thereby diminishing the resulting density gradients, their interactions with desired convection, and their contributions to unwanted buoyancy-driven convection. One possibility is to lower gravity.

For certain scientific measurements and special processing of materials, it is required to hold a liquid or, rarely, a solid sample away from any container walls and the various sorts of contamination and interference that walls bring. The sample must be levitated with its weight offset by another force: electromagnetic, aerodynamic, or acoustic. Owing to side effects that involve surface tension as well, the capability of any one of these alone is limited; their use in combination has scarcely been explored. However, alone or in concert, these forces cannot be brought to bear on a levitated liquid sample without causing electromagnetically driven or shear-driven convection. The resulting flow velocities are, for a given viscosity level, roughly proportional to the levitating force and thus to the weight of the sample, which in turn varies as the sample volume and density and the magnitude of gravity. One of the ways of slowing convection is to reduce gravity, but this may merely uncover Marangoni convection from the liquid meniscus that is necessarily present. Moreover, strong convection is sometimes wanted, for example, during mixing of glass samples to achieve uniform composition and eliminate bubbles or during growth of semiconductor crystals to prevent radial segregation of a dopant.

#### CONTROL OF CONVECTION IN MATERIALS PROCESSING

Many means of controlling convection are evident from the preceding discussion. When convection is deleterious, it can be reduced by raising the relative importance of viscosity, most readily by altering the configuration or reducing the size of the system. In some cases, it can be opposed by applying a static magnetic field to induce velocity-dependent electromagnetic damping forces. However, such forces seldom damp convection uniformly and, if flow occurs, are accompanied by ohmic heating that can compound the driving forces for convection. In other cases, the force driving the convection can be diminished or eliminated. In the case of natural convection, the underlying density non-uniformity can rarely be avoided, but sometimes by equipment design and operating procedure it can be organized into a *stable* stratification of horizontal fluid layers that can actually eliminate buoyancy-driven convection. This requires a *steady* gravitational field and so is impossible in an orbiting spacecraft. A related scheme is to reduce the consequences of convection rather than convection itself, for instance, by aligning the flow direction at nearly right angles to the concentration variation in critical places. If one particular type of convection, for example, natural convection or Marangoni convection, is deleterious, it can in some cases simply be buried in much stronger forced convection that is easier to characterize and control.

These means, among others, have long been employed to cope with buoyancy phenomena that arise from the action of gravity in continuous matter. But the more complex the physical processes involved, the fewer options scientists and engineers have for controlling buoyancy-driven convection throughout a system. Whether or not it can be satisfactorily accommodated at one g often depends on the experimenter's understanding and design ingenuity, background and experience, breadth of view and motivation. When modification of gravity is indicated, a variety of means is available. They include increasing the effective gravitational field (and accepting fluctuations in its magnitude and direction) by using a centrifuge or decreasing the field by using a drop tower, aircraft, or rocket in free-fall trajectory or, for longer periods, a spacecraft in orbit.

#### POTENTIAL ADVANTAGES OF LOW GRAVITY

On the basis of the information set forth above, the experimental capabilities offered by a sustained low-gravity environment appear to be the following.

*The possibility of reducing or practically eliminating buoyancy-driven natural convection for substantial periods of time.* As has been shown, there are many technologically important, scientifically challenging processes sufficiently complex that the effects of buoyancy are obscured or cannot be controlled independently of other phenomena. Nor can these processes be modeled mathematically without numerous simplifying approximations that warrant experimental testing. Especially where natural convection is believed to be deleterious, experiments conducted in low gravity *may be* of some use in making a process more understandable and in stimulating earth-bound developments. Such experiments may reveal other convection phenomena that ordinarily are masked by natural convection. The possibility of obtaining a product having uniquely useful properties in such an experiment cannot be excluded.

*The possibility of testing experimentally the basic assumptions necessary in theoretical models of systems in which complicated patterns of fluid density variation are inherent.* There are fundamental physical processes such as solidification and combustion that couple transformation and transport phenomena and unavoidably generate both density gradients and density-gradient-driven convection. Density gradients are complicated in that they never permit total stable stratification against buoyancy-driven natural convection in the earth's gravity. Natural convection tends to interfere with planar, spherical, or other simple symmetries in those experiments by which basic physical theory can most incisively

be tested and directed. This is particularly important when, as is common of complex nonlinear phenomena, theory predicts that a system may behave in more than one way. In these circumstances, careful experiments in low gravity can advance scientific understanding.

*The possibility of reducing or practically eliminating the settling of particles for substantial periods of time.* This potential advantage also arises from suppressing buoyancy phenomena. It may be useful for scientific or technological purposes.

*The possibility of levitating and isolating larger samples for containerless processing.* There are property measurements and basic processes for which it is advantageous or necessary to isolate a liquid sample from container walls, and there are practical limits on sample sizes that can be levitated on earth. Furthermore, levitation is accompanied by convection. Low gravity has the potential advantage of allowing levitation of larger samples. This may be partly offset by problems of positioning and manipulating the sample in a fluctuating background gravitational field and, if acoustic positioning is used, of contending with a surrounding gas. The possibility exists of obtaining exemplary materials having unique properties by containerless processing in space.

## INFLUENCES OF GRAVITY ON MATERIALS PROCESSING

The previous chapter described the elements of the space environment, principally the virtual absence of gravity, that might affect processing of materials. In this chapter, several generic processes are discussed in the light of this information. The Committee chose to emphasize phenomena rather than classes of materials, in the belief that this approach would be more manageable and useful than a categorical review of materials. The study therefore dealt with certain general phenomena, notably solidification, electrophoresis, and combustion, and with certain process methods such as containerless and droplet processing.

### SOLIDIFICATION

Solidification of materials from the melt or solution invariably involves convection. Fundamental study and application of solidification processes therefore require an understanding of convective transport of matter. It may be necessary in certain cases to control convection in order to produce better materials. This may be done by reducing convection or by producing a mode of convection sufficiently simple or regular that it may be characterized and controlled.

The optimum would be to reduce fluid flow to that relative motion of phases necessary to accommodate density changes on solidification. For unidirectional solidification with a solid-liquid interface of invariant shape,\* such a flow would be a spatially uniform linear motion of the liquid relative to the solid. Such a liquid could be described as a convectionless system, provided one employs a reference frame (perhaps not the laboratory frame) in which the liquid is at rest. Solidification under these idealized conditions, which may be referred to as

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\* Solidification of a sphere of constant density from an infinite medium would require a radial flow whose magnitude is inversely proportional to the square of the radial coordinate. For more complicated and/or non-steady-state solidification, more complicated flows are necessary just to accommodate density differences between solid and liquid.

solidification under conditions controlled only by diffusion, could provide a meeting ground for theory and experiment. This would permit qualitative comparison, provided edge effects could be made negligible. This, in turn, could advance quantitative understanding of the solidification process, contribute to the measurement of basic material properties such as distribution coefficients and diffusion coefficients, and possibly lead to the production of more nearly perfect crystals.

Some ways to investigate the degree to which these idealized conditions can be achieved in actual systems are containment on earth in fine capillary tubes to promote strong viscous damping; favorable orientation relative to the gravity vector to achieve stable stratification in the face of non-uniform density; electromagnetic damping for electrically conducting melts by application of static magnetic fields; and reduction of the gravitational field by achieving a state of free-fall.

Unidirectional solidification in fine capillaries is an effective means of reducing convection but is not without complications that arise because of thermal and kinetic interferences from the walls; hydrodynamic boundary layer convection at the wall; meniscus effects associated with the solid-liquid-container triple junction; capillarity effects as the capillary bore becomes small; and enhanced tendency for contamination because of the large surface-to-volume ratio.

Favorable orientation by solidifying either upward or downward to achieve stable configurations in systems of non-uniform density is effective for plane-front growth and practical if the favorable direction is upward. If downward solidification is necessary to reduce convection, the usual volume shrinkage accompanying the liquid-to-solid transition will necessitate some stratagem to maintain the liquid in contact with the solid, which may stick to the container. For non-planar growth, orientation may not be sufficiently effective to achieve stable configuration. Moreover, if the gradient of more than one field, such as a temperature and a composition, affects density, it may not be possible to eliminate the resulting double diffusive instabilities.

Use of electromagnetic damping by a static magnetic field is limited to materials that are fairly good electrical conductors, if the strength of the magnetic field required is to be within practical limits. Moreover, damping occurs only for fluid motions perpendicular to the magnetic field. It is fairly easy to damp rapid random convection using easily attainable magnetic fields. Damping of laminar convection is more difficult and requires very intense fields. Compatibility between thermal requirements and large magnetic fields may necessitate very small sample sizes. In some cases, magnetic damping may be impossible at very high temperatures.

Reduction of buoyancy-driven convection by going into a low-gravity environment is attractive because it could permit working with relatively large samples, and one would not be limited to electrically conducting

materials or to low-temperature systems. It is important to note, however, that convection will not be entirely eliminated in low-gravity experiments, not only because of the influence of the residual gravity, but also because of secondary driving forces such as surface tension gradients that give rise to Marangoni flow (as explained in the preceding chapter). It is particularly important, then, that experiments in space that count heavily on reduction in convection be adequately supported and complemented by ground-based research as well as by experiments in space specifically directed at the study and demonstration of convective flows.

## SPECIFIC EXAMPLES OF SOLIDIFICATION EXPERIMENTS

Several examples of types of solidification experiments that might be conducted in a low-gravity environment are examined in this section. The list is not intended to be complete, but rather to illustrate some of the basic considerations involved.

### Plane-Front Growth of Single-Phase Solids

If plane-front growth of single-phase solids can be achieved under conditions that sufficiently approximate diffusion-controlled growth, it should be possible to compare the resulting composition profile with that predicted by one-dimensional theory. The segregated region of the liquid will be a mass transfer, or diffusion, boundary layer\* ahead of the solid-liquid interface. Results are likely to permit determination of values of diffusion coefficients in the liquid as well as equilibrium and non-equilibrium values of the distribution coefficient\*\* that determines the amount of segregation on solidification. Product crystals are likely to be more nearly perfect because of the absence of compositional banding caused by convection-induced temperature fluctuations and concomitant fluctuations in growth rate. Some evidence that this is possible is provided by experiments using indium antimonide on Skylab (Appendix, experiments M 560, M 562 and M 563), and using germanium on Apollo-Soyuz (Appendix, experiments MA 060 and MA 150).

### Plane-Front Growth of Polyphase Solids

Plane-front growth of composites from off-eutectic melts is a possible candidate for space experiments. However, growth of binary eutectics from liquids of exactly eutectic composition should not be severely affected by convection because the diffusion boundary layer is very small (of the order of the lamellar spacing) and is likely to be well within

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\* Of thickness of the order of  $D/V$ , where  $D$  is the interdiffusion coefficient and  $V$  is the growth velocity.

\*\* The ratio of the concentration in the solid to the concentration in the liquid from which it freezes.

the momentum boundary layer. There might, nevertheless, be benefits from reduction of growth-rate fluctuations. For coupled growth of composites from off-eutectic melts or in ternary systems, however, the diffusion boundary layer will be of the order of  $D/V$ , as for single-phase materials, because long-range diffusive transport is required. In such cases, the low-gravity environment might lead to important results for the same reasons as for single-phase solids.

#### Mixed Composites

The possibility of using a low-gravity environment to produce uniformly dispersed mixed composites having phases of different density seems at first to be attractive. One might, for example, attempt to solidify a system with a miscibility gap in the liquid or, alternatively, to mix particles or fibers of solid within a liquid and solidify the entire mass. However, it appears that for very small particles, earth-based processing methods are sufficient because the agglomeration of very small particles is governed by Brownian motion and therefore is not substantially influenced by a gravitational field. On earth, larger particles, for which gravity segregation is important, can be mixed with partially solidified materials, thereby restricting segregation in the solid. Furthermore, there exist immiscible liquids with isodensity that can be studied on earth to gain fundamental knowledge. The Committee's conclusion is that space processing offers no advantages in this area.

#### Large Mono-Disperse Particles

If large mono-disperse particles could be prepared in quantity, they would have important uses. Their preparation on earth is impeded by sedimentation. In a low-gravity environment, the absence of strong convective flow may permit isotropic and homogeneous growth of nucleated mono-disperse particles throughout the volume of the liquid latex from which they are made. It may therefore be useful to explore the possibility of preparing large mono-disperse particles by experiments in space.

#### Non-Planar Growth

Situations in which the solid-liquid interface is non-planar (cellular or dendritic) are so difficult to analyze that to date all known theoretical solutions pertain only to conditions where diffusion predominates. For these complex growth forms, convection on earth is multidirectional and hence difficult to eliminate by orientation relative to gravity or application of a magnetic field. Because there are many gaps in fundamental understanding of these complex growth forms, it would be useful to make measurements at low gravity of dendritic and cellular growth rates and shapes using a low-gravity environment to reduce convection.

## CONTAINERLESS PROCESSING

Avoidance of a container may be an important factor in processing of materials. Harmful effects associated with containers may include chemical contamination of the melt, structural contamination of the melt (e.g., crystal nuclei), and limitation on the chemical environment or the temperature to which the melt can be taken.

Isolation of materials in terrestrial work is commonly achieved by levitation. Several techniques for levitating small melts have been developed to some degree. Of these, the best known is electromagnetic levitation, which has been successfully used for small melts of electrically conducting materials. Disadvantages include the fact that the levitating force is inherently coupled with heating so that independent control of these parameters is not possible. Other techniques for levitating liquids in the presence of gravity, not confined to electrically conducting materials, include the use of acoustic standing waves and of gas streams. In all earth-based techniques, there is in practice an upper limit to the size of melts that can be levitated. The problems of levitation may be eased by recourse to a low-gravity environment. The low gravity achievable in orbiting spacecraft will still subject materials to small forces, as described in the preceding chapter. Some modest positioning force, either electromagnetic, acoustic, or aerodynamic, will often have to be applied.

The following discussion of containerless processing, while not comprehensive, includes some of the uses that have come to the attention of the Committee.

### Preparation of Glasses and Ceramics

Glass melts are highly reactive materials. They react somewhat with virtually any container, slightly with most materials, but sometimes appreciably in the case of silicate glasses, even with platinum. The degree of reaction and subsequent contamination is sufficiently small to be tolerable for ordinary commercial glasses. However, special applications, such as high-power laser systems for fusion experiments, require glasses with very low levels of contamination. The use of containerless methods, at least to obtain exemplary glasses for research, has some value and may benefit from a low-gravity approach.

Glasses are formed when the cooling rate of the melt is sufficiently rapid to prevent appreciable nucleation and crystal growth. Uhlmann\* has given a theoretical treatment for one-component glasses and shown how to prepare temperature-time transformation curves that show the time required at a chosen temperature for homogeneous nucleation and growth

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\* D. R. Uhlmann, "A Kinetic Treatment of Glass Formation." *Journal of Noncrystalline Solids* 7(1972):337-378.

to produce a given small fraction of crystalline phase. Neilson and Weinberg\* have calculated transformation times for laser glass compositions with high CaO content. These glasses are desirable for their increased laser efficiency but they cannot now be made on earth because the high CaO content causes them to nucleate heterogeneously by contact with the container. Crystal growth then occurs at a high rate just below the melting point. If heterogeneous nucleation can be avoided by containerless processing, new families of glasses with desirable properties could advance optical technology.

Ceramics must be prepared by sintering at such high temperatures that they are almost invariably contaminated by the container in which they are made. Containerless processing may offer valuable research opportunities for the preparation of high-purity ceramics.

#### Thermodynamics and Kinetics

Knowledge of high-temperature thermodynamics and chemical kinetics is fundamental to the design of high-temperature materials processes. Studies of reactive liquids at high temperatures have been limited by contamination from the container. Thermodynamic quantities not now known for many materials include enthalpies, specific heats, heats of fusion, and densities.

Substances of interest for containerless thermodynamics studies include virtually every liquid above approximately 1000°C. This includes even electrically conducting liquids that have been studied to some extent using electromagnetic levitation because the studies have been hampered by the fact that temperature cannot be controlled independently of the levitating force. Such studies systematically carried out over a wide range of temperatures have not generally been possible. Examples of substances awaiting good thermodynamic measurement at high temperatures include pure silicon and refractory oxides, sulfates, carbides, and nitrides. Development of containerless heating and levitation techniques for reactive melts could be very useful for the determination of high-temperature phase equilibria.

#### Purification of Materials

In addition to the use of containerless processing to avoid contamination, there is the possibility of using containerless processing for purification. Purification with respect to elements more volatile than the host material can, in principle, be done by simple evaporation, provided the melt is stirred. The theory has been developed in detail

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\* G. F. Neilson and M. C. Weinberg, "Outer Space Formation of a Laser Host Glass." *Journal of Noncrystalline Solids* 23(1977):43-58.

and includes the capability to deal with systems undergoing reaction while evaporating.\*

## BIOLOGICAL SEPARATIONS AND ELECTROPHORESIS

Cells of living organisms and macromolecules associated with biological processes almost always occur in complex arrays in mixtures and in the presence of water and a virtual infinity of small molecules and ions. Many studies of the identities, structures, and properties of these cells and macromolecules depend on adequate separation of the species of interest from other species. The characterization of plasma proteins by Tiselius in the 1930s, for example, hinged on the discovery of a method, termed *electrophoresis*, for discrete separation of these molecules. Electrophoresis is based on the motion of charged particles in a fluid under the influence of an applied electric field. Separation of components of a mixture results from differences in the electric charge and the size of the particles that produce differences in the mobility of macro-ions. Electrophoresis has been applied over the years to a number of problems in the separation of cells and macromolecules. For reasons outlined below, some investigators believe a low-gravity environment has potential benefit to electrophoresis. Because this possible use of space facilities had aroused some expectations for progress in medicine, the Committee considered at some length biological separations and cell electrophoresis.

In the presence of the earth's gravity, electrophoretic forces are disturbed by convection. Suppression of convection is usually achieved in terrestrial apparatus by constraining the internal radius or thickness of the separation apparatus, but then the processing rate is also limited. In some apparatus, resolution can be increased by raising the voltage gradient, but joule heating from the passage of current then increases the problem of thermal convection. This heating problem is commonly countered by restricting the ionic concentration in the sample solution, but for solutions containing living cells, such ionic concentrations usually are undesirably low for maintenance of physiologic activity.

Elaborations of apparatus design or operation have been invoked to suppress convection or sedimentation. Some of these arrangements have been used, others have so far only been hypothesized. In the absence of extensive analysis of electrophoretic flow, experimenters have proceeded, often with great ingenuity, to design around the difficulties arising from the basic constraints. Certain compromises have knowingly been made but, to the best of the Committee's ability to determine, electrophoretic apparatus for use on earth has not yet been optimized.

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\* R. C. Paule, "Calculation of Complex Equilibria Involving Vaporization into Vacuum." *High Temperature Science* 6(1974):267-275.

## Utility of the Space Environment

Some investigators claim that elimination of convection will lift the restriction on the dimensions of the chamber and permit use of wider chambers with increased production rates. For separation of living cells, freedom from convection would permit use of a smaller voltage gradient and therefore use of solutions of high conductivity, approximating physiological tonicity. However, the resolving power of existing electrophoresis apparatus on earth appears to be greater than that needed to separate the cell mixtures already known. On the subject of production rate, some investigators consulted by the Committee predicted enhancement factors of 30 to 100 in a low-gravity environment. These figures have not had the benefit of much theoretical or experimental support. Moreover, the estimate is in dispute; maximum enhancement in space by a factor of two or three appeared to others to be more likely.

The uses of terrestrial electrophoresis in non-routine separations have not yet been sufficiently explored to provide the proper basis for investigations in low gravity. The results of earlier experiments in electrophoresis in space (see Appendix) are tenuous and the Committee considers it important that the effects of varying significant parameters in electrophoretic systems be investigated by earth-based studies. The objective of learning more about how electrophoresis apparatus should be designed and how gravity may affect the electrophoretic process will best be answered through well-planned terrestrial research rather than experiments in a low-gravity environment. In terms of what is now known, the Committee concludes that there is no pressing need for an enlarged trial of electrophoresis in space.

## DROPLET PROCESSING

An aerosol is, in effect, an assembly of micro-containers, each of which can be used as the site of a reaction or process. The micro-droplets function as containers without solid walls by taking advantage of the surface tension of the droplet. In biology and medicine, the need to carry out many different types of processing simultaneously in many separate containers is rather common. It is conceivable that a similar need for micro-containers may develop in the physical sciences and engineering, especially if composition or reaction conditions within each drop can be measured and made different from those in other drops.

The droplet technique is already being experimented with as a means for isolating single cells and then permitting the cells to multiply. At normal gravity, however, an aerosol composed of water droplets of 12  $\mu\text{m}$  diameter persists for only a few hours. In biological systems, however, it is often necessary to process larger volumes for longer times. For example, in antibiotic production, it could be useful to suspend drops of 50  $\mu\text{m}$  to 100  $\mu\text{m}$  for 50 to 100 hours. An aerosol at low gravity could persist indefinitely, provided that collisions among the droplets and of droplets with walls could be avoided. To take advantage of such

an assembly of micro-containers, techniques are needed to generate the micro-droplets, each with a characterized composition. Also important are controlling velocities and positions and reducing mechanisms of interchange of matter among the droplets. Careful consideration will be needed to determine if the advantage of  $10^6$  to  $10^7$  long-lived micro-containers at low gravity would justify, for some specific problem, the difficult control and analytical techniques that must be developed. It appears to the Committee that the possibilities of droplet processing warrant exploration.

#### COMBUSTION\*

Combustion is closely linked in science to thermodynamics, chemical kinetics, and fluid dynamics and it is coupled in engineering to energy conversion, flammability of materials, environmental quality, and home and industrial safety. In terrestrial combustion, buoyancy-driven convection plays a major if not dominant role. A question arises as to how other factors such as chemistry and transport fundamentally affect combustion. While these factors can be treated individually through use of fundamental chemistry and computer modeling, study of combustion phenomena may benefit from removal of gravitational influences.

In addition to convection, many other complex phenomena arising from evolved heat are involved in a combustion process. Some may be characterized by macroscopic quantities such as ignition energies, auto-ignition conditions, and limits of flame existence. While a large body of data on fuel-oxidizer systems has been accumulated, it is not possible to test all combinations. At present, combustion theory depends heavily on the ability to construct models and conduct experiments that vary or isolate certain parameters. With continuing growth in the power of computers, the ability to model complex systems is also increasing. Concurrently, ability to perform highly detailed experiments on combustion systems with powerful tools such as tunable lasers is steadily improving.

#### Burning of Single Droplets

The simplest diffusion-controlled system -- burning of single droplets -- is a basic approach for studying combustion processes. Droplet-burning models, which have long been in use, are based on certain assumptions, for example, that the droplet surface and surrounding flame front are located on concentric spheres. Ground-based observations of droplet burning, however, show that luminous flame boundaries are, in fact, far from spherical and are actually determined by convection currents. Early experiments on burning droplets falling freely in earth-based chambers succeeded in simulating droplets burning with spherical symmetry in the absence of convection effects. However, the test duration of about one

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\* This section draws heavily on a paper prepared for the Committee by S. S. Penner.

second did not permit achieving steady-state conditions. Using taller drop towers, which provide two to five seconds of free fall, these experiments on single droplets have been extended. Nevertheless, free-fall duration in drop towers is marginal. Low-gravity experiments in space on the burning of single droplets can provide data on parameters such as symmetry, flame radius, and droplet radius and size limits at extinction.

#### Burning of Multiple Droplets

Interference during burning of two or more droplets in close proximity is dominated by convection currents. Flame boundaries merge when droplets are sufficiently closely spaced. In the absence of convection, interference between droplets during burning may be expected to be basically different in character from that observed in ground-based studies.

#### Burning of Droplet and Particle Clouds

Flame propagation rates and extinction conditions in the burning of droplet and particle clouds are strongly influenced by convection. Examination of fuel clouds should stress determination of parameters such as average flame propagation speeds as functions of mean drop size and drop-size distribution. Experiments in space could provide data on temperature and composition fields for near-limit flames, flame shape and speed, and extinction limits.

Some preliminary work in particle-cloud burning has been done in drop towers, but there are severe problems in trying to prepare stationary pre-mixed particle clouds in the presence of gravity. An adequate elaboration of cloud-burning experiments suitable for space-based observations remains to be made. Ground-based work to define potential space experiments should properly start with careful formulation of the applicable conservation equations.

#### Laminar Flame Propagation

Theoretical description of one-dimensional laminar flame speeds in pre-mixed gases, with proper application of known reaction mechanisms and rates, has served in the evolution of combustion science. The presence of convection phenomena always influences the determination of these fundamental parameters in experiments conducted in ground-based laboratories.

#### Flammability Limits

An important consideration in understanding combustion is the boundary of composition between what is burning and what is not yet burning, that is, the flammability limit. The importance of buoyancy effects on flammability limits is shown by the contrast in the observed limiting mixture compositions for upward and downward flame propagation. A flat

flame propagating downward is dynamically stable because the hot, lighter combustion products are produced above the heavier combustible mixture. The reverse situation prevails for upward flame propagation in which buoyancy effects are relatively more important. Observed flammability limits are wider for upward than for downward flame propagation. However, flame propagation is not observed in ideal, one-dimensional systems. The presence of walls produces cooled gas layers at the boundaries and destroys the simplified dimensionality of the problem. As a result, it is not clear whether buoyancy plays a role in defining flammability limits for downward flame propagation. A few well-designed tests to examine flammability limits for freely propagating near-limit systems under conditions of low gravity appear warranted.

The Committee concludes that certain classic model systems and certain aspects of the combustion process can be usefully studied with the virtual elimination of convection. These include burning of single droplets, multiple droplets, and droplet and particle clouds; laminar flame propagation; and flammability limits. While such studies are expected to contribute to understanding combustion processes, it is not likely that they will be useful in determining primary factors such as chemical reaction rates or transport processes.

## A PERSPECTIVE ON THE TECHNOLOGY OF PROCESSING MATERIALS IN SPACE

The previous chapter outlines some of the fundamental aspects of processing materials in space. It also refers to a number of technologies without addressing the commercial aspects of those areas. Commercial analyses are more properly the domain of a separate study. The role of technology in the space environment is, nevertheless, closely linked to earth-based commercial aspects. The Committee therefore examined as an example the possible importance of growing semiconductor crystals in space for commercial use. This case seemed particularly appropriate to examine because it also involves solidification and containerless processing, matters that the Committee examined in detail. Furthermore, data were available to make possible consideration of the comparative costs of producing crystals in space and on earth.

Among the advantages claimed for growing semiconductor electronic crystals in space are improved homogeneity, greater purity, reduction of the number of physical defects and imperfections, ability to grow large-diameter crystals, and ability to grow crystals as flat ribbons.\* Two assumptions are implied: first, that semiconductor crystals have stringent requirements for purity, uniformity, and perfection; second, that the availability of electronic materials with fewer imperfections or greater purity will permit making electronic devices with improved characteristics. It has been further assumed that the improvements in device performance would justify growing crystals in space even if unit costs were increased.

In considering the validity of the assumptions, the following questions must be answered:

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\* The interest in flat ribbon crystals is, in turn, based on an assumption that the availability of flat ribbon would eliminate the cost of sawing wafers from cylindrical material and polishing the wafers and that the cost of preparing a chip is independent of wafer size.

Is it true that crystals of materials most commonly used for electronic devices -- silicon, III-V compounds, garnets -- grown in space will be physically and/or chemically superior to those grown on earth?

Even if the crystal material is superior, does it necessarily follow that a device incorporating the superior material will have improved properties?

If the device has improved properties, will the improvement in performance of the device have any significant positive effect on the performance of the system using the device?

If the performance of the system is improved, is the improvement worth the additional cost arising from manufacturing the starting material in space?

Can crystals of significantly larger diameter be grown in space?

Can flat ribbon crystals be grown in space?

These questions are addressed in the following discussion.

#### POSSIBLE ADVANTAGES OF GROWING CRYSTALS IN SPACE

Experiments on Skylab by Wiedemeier, Witt and Gatos\* have been cited as evidence that crystals grown in a low-g environment are superior to those grown on earth. The Wiedemeier experiments involved growing GeSe and GeTe crystals from vapor in a closed system using  $\text{GeI}_4$  as the transport agent. A direct comparison was made between mass transport rates in space and on earth. The Witt-Gatos experiments involved melting and

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\* H. Wiedemeier et al., "Crystal Growth and Transport Rates of GeSe and GeTe in Microgravity Environment." Journal of Crystal Growth 31(1975):36-43; H. Wiedemeier et al., "Vapor Growth of GeSe and GeTe Single Crystals in Micro-gravity." Proceedings: Third Space Processing Symposium, Skylab Results, Vol. 1, NASA M-74-5, George C. Marshall Space Flight Center, Alabama, pp. 235-256 (June 1974); A. F. Witt et al., "Crystal Growth and Steady-State Segregation under Zero Gravity:  $\text{InSb}$ ." Journal of the Electrochemical Society 122(2)(1975):276-283; A. F. Witt et al., "Steady-State Growth and Segregation under Zero Gravity:  $\text{InSb}$ ." Skylab Results, Vol. 1, pp. 275-288.

resolidifying a portion of a Te-doped InSb crystal originally grown on earth. Witt and Gatos repeated the experiments on earth, thus making possible a direct comparison of earth-grown to space-grown crystals. In contrast to much of the materials processing work in space to date, careful ground-based work was done in both these cases.

In Wiedemeier's experiments, crystals grown on earth showed distorted surfaces of platelets and needles. Aggregation and twinning were frequently observed. Nearly all octahedral crystals grown on earth revealed partially hollow growth habits while the corresponding space-grown crystals had considerably more compact structures. The faces of space-grown crystals showed a higher degree of smoothness and crystal-line perfection; the edges were better defined. These improvements were attributed to the absence of gravity-driven convection and to growth under conditions controlled only by diffusion. A second observation was that the mass transport rate was greater than expected in a low-g environment. The reason for this is not understood. Unfortunately, the earth-based experiments used a horizontal orientation of the apparatus, but neither of the two possible vertical orientations.

In the Witt-Gatos experiments, the objective was to achieve growth under diffusion-controlled, steady-state conditions. They have reported to the Committee that these growth conditions were obtained, leading to three-dimensional microscale chemical homogeneity over macroscale dimensions. They also reported a phenomenon never previously observed and not predicted theoretically that they attributed to surface-tension effects, that is, the Te-doped melt did not wet the quartz wall of the container but solidified with a free-surface (unconfined) configuration. Under forced-contact conditions, intimate contact between the melt and confining walls was prevented, and the growing crystal system was essentially isolated from its container by the formation of narrow surface ridges. The overall conclusion of the investigators was that the InSb experiment proved unambiguously the uniqueness of low-gravity conditions for obtaining fundamental data on crystal growth and segregation associated with solidification and that the results demonstrated advantages of processing materials in space.

The results reported for these crystal-growth experiments appear to be valid. Adequate and carefully conducted earth-based experiments were performed for comparison. Under the particular growth conditions used, improvements in both physical and chemical properties were obtained. However, in the Committee's opinion, these results are not sufficient to permit concluding whether growth in space of crystals for commercial electronic devices is or is not viable.

Neither of the materials studied in the vapor-growth experiments, GeSe or GeTe, is expected to be used in practical electronic devices. Moreover, the growth technique used -- closed-tube vapor transport -- is not expected to be used for growing those materials that do have significant practical applications. Currently, vapor growth is used to

prepare GaAs structures for a variety of important microwave devices (e.g., Gunn oscillators, IMPATT diodes, and varactors) and to prepare ternary or quaternary III-V materials for light-emitting devices (e.g., diodes for displays and lasers). In these cases, an open-tube flow system is used, a system in which the particular effects observed in the closed-tube system in low gravity would probably not be observed.

Neither is the InSb grown by Witt and Gatos now commercially important, for it is used only for a small number of specialized infrared detectors. The growth method used by Witt and Gatos -- directional solidification in a closed tube -- is not used for production of commercial electronic devices. It is impossible to extrapolate the results of the InSb, GeSe, or GeTe experiments to specific materials or processes used or planned for use commercially or to predict any specific advantages of processing those materials in a low-g environment.

The only experiments in space using a current production process were those involving the float-zone method. The experiments used rudimentary equipment and no effects were observed that could be used to predict improved properties for material grown in space.

#### EFFECTS OF IMPROVEMENTS IN THE QUALITY OF STARTING MATERIAL ON THE PERFORMANCE OF ELECTRONIC DEVICES

It has been said that better starting material leads to better device performance, but, in fact, the quality of starting material is not the limiting consideration for most devices presently manufactured. Even if starting material were perfect, most fabrication processes for devices involve steps at high temperatures that induce physical and chemical defects far in excess of those originally present. Some present devices and some in the development stage are, however, clearly affected by the quality of presently available material. Others are probably affected, but the correlation is not clear. In this discussion, materials considered will be restricted to those used, or expected to be used, in relatively high volume, specifically silicon, GaAs, III-V alloys, and garnets.

Silicon is the most widely used material of the electronic semiconductor device industry. It is used in a variety of devices from discrete transistors, rectifiers, diodes, and radiation sensors to complex integrated circuits containing several thousand devices on a chip. These devices are formed on slices of single-crystal silicon, bulk-grown by the Czochralski or the float-zone technique or in epitaxial layers formed by chemical vapor deposition on bulk slices.

Many types of physical and chemical imperfections in silicon can affect device performance, yield of usable chips from the parent material, or both. These include point defects and clusters of point defects, heavy-metal impurities that cause deep levels and traps, carbon and

oxygen, and non-uniform distribution of added doping agents. These imperfections influence properties of the silicon such as uniformity of resistivity, susceptibility of resistivity to changes during heat treatment, net dopant concentration, minority-carrier lifetime, and carrier mobilities. These in turn can cause a wide variety of problems in devices, such as reduced breakdown voltage, non-uniform heating, non-uniform switching, increased leakage currents, and reduced and non-uniform transistor gain. They can also cause differences among devices fabricated simultaneously on the same slice, thereby reducing the yield.

For most silicon devices, defects introduced during processing, particularly during photolithographic steps or high-temperature operations, far exceed those in typical starting material. As processes are improved through new technology, the quality of the starting material will become more important.

Present commercial silicon is not very uniform. Across a slice, resistivity variations of as much as 30 percent for N-type and 15 percent for P-type are not uncommon. The degree of physical perfection also needs to be improved; for example, so-called dislocation-free silicon typically contains swirl defects. Some problems are amplified significantly by contamination and lack of care during typical mass production growth of silicon crystals.

Growth under low-g conditions might help solve some of these problems; for example, the Witt-Gatos experiments indicate doping uniformity might be improved. However, earth-bound processes can almost certainly be improved significantly by further research and development. For example, the relatively new technique of neutron irradiation to create a desired dopant concentration by transmutation of silicon to phosphorous gives N-type material with a very high degree of uniformity. Unfortunately, industry R&D in silicon-crystal growth processes has been extremely sparse for several years, a clear indication that of the many problems in developing new and improved electronic devices, those associated with the starting material had low priority.

Another commercially important group of electronic materials is based on GaAs and its ternary and quaternary alloys with other group-III and -V elements. Microwave devices generally use vapor growth to form the required two or more very thin layers of different dopant concentrations. Uniformity of the dopant concentration and an abrupt transition between the regions is very important, as is surface morphology for some devices. Important improvements are being made in the ability to grow these complex devices but significantly better devices could be obtained if better material were available. The growth processes are complicated and sophisticated, and considerable attention and intervention by a highly trained operator is necessary during growth. Unless the processes can be greatly simplified and automated, and unless growth in low gravity is shown to give significant improvement in the material, it is doubtful that it will be advantageous to perform the process in space.

Light-emitting diodes are fabricated in III-V alloys such as (Ga, Al)As and Ga(As, P). Materials defects reduce the efficiency for converting current into light. It is not possible, however, to predict how significant would be the gain from using perfect materials. No experiments on liquid epitaxy have been conducted in space, so the potential for improving epitaxial materials by growth in low gravity is not known. Solid-state lasers are also fabricated by epitaxial deposition of successive layers of III-V materials. In this case, the advantage of more perfect material would be to prolong the operating life of the devices.

Garnet crystals, particularly those based on rare earth elements, are becoming increasingly important for use in magnetic bubble memory devices. Physical perfection is of great importance in these devices. Chips are relatively large (up to 1 cm square) and a single defect can make a serial shift-register inoperative. Predominant defects, usually at a density of 2 to 5 per square centimeter, are dislocations and inclusions from the container. It is possible on earth to grow crystals completely free from such defects by using great care. Even though perfection requirements are very stringent, most experimenters believe that adequate quality can be obtained on earth and that space processing will not yield any advantages.

#### ECONOMICS

An A. D. Little study\* in 1974 suggested a possible economic advantage arising from a potential increase in diameter for crystals grown in space. A. D. Little has recently re-examined the matter\*\* and the conclusion now is that there is no economic advantage. A study carried out for NASA by McDonnell Douglas\*\*\* suggests that there are possible economic advantages for growing silicon in space for fabrication into integrated circuits on earth. The McDonnell Douglas study assumes a hypothetical system for growing silicon in ribbon form and an advantage arising from increased yield in fabrication into devices leading to the lower cost. A yield improvement factor of 4.5 is postulated, 60 percent

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\* A. A. Fowle et al., Float-Zone Processing in a Weightless Environment. NASA CR-143876, George C. Marshall Space Flight Center, Alabama, October 1974.

\*\* A. A. Fowle et al., Float-Zone Processing in a Weightless Environment. NASA CR-2768, George C. Marshall Space Flight Center, Alabama, November 1976.

\*\*\* McDonnell Douglas Corporation, Feasibility Study of Commercial Space Manufacturing. NASA Contract NAS 8-31353, St. Louis, Missouri, 1975.

of which is due to eliminating sawing and polishing the silicon. The remainder is attributed to lower losses due to greater structural perfection, more uniform resistivity, fewer dopant defects, and geometrical advantage (rectangular vs. circular). The McDonnell Douglas system also assumes that a low-g environment will make ribbon growth feasible. It would be beneficial to grow silicon in ribbon form with sufficiently perfect surfaces to eliminate polishing. A large R&D effort has been devoted to developing an earth-based system for doing this. The primary barrier to growing satisfactory ribbons on earth has been problems associated with the guides used for obtaining the required shape and size. The same problem will exist in space; it is not obvious how eliminating the gravity force will solve the problem.

#### IMPACT OF SPACE EXPERIMENTS ON GROUND-BASED GROWTH OF ELECTRONIC MATERIALS

Methods now used for the commercial growth of large crystals of electronic materials such as silicon have been developed empirically with little theoretical understanding of what occurs at the microscale level during growth. It has been argued that much better theoretical understanding of the growth process can be obtained by experiments in low gravity and that such knowledge would lead to significant improvements in earth-based crystal growth. Certainly, the Witt-Gatos experiments on Ga-doped germanium (Appendix, experiment MA 060) have demonstrated that segregation in space-grown crystals can be understood quantitatively in terms of a model in which convection is absent. Experiments of this kind are bound to yield useful fundamental information such as distribution coefficients, diffusion coefficients, and a better understanding of transient effects and radial segregation. Although this information will undoubtedly lead to an improvement in the growth of crystals on earth, the significance of such an improvement for device applications remains unresolved.

## DEFINITION, ORGANIZATION, AND ADMINISTRATION OF A VIABLE PROGRAM

If a program for processing materials in space is to be viable, the program itself and its associated facilities must be properly defined, organized, and administered. This chapter conveys the Committee's views on these matters.

### PROGRAM DEFINITION

Federal agencies such as the National Science Foundation, Department of Energy, National Institutes of Health, and Department of Defense have responsibility to fund those aspects of research in materials science and technology germane to their missions. In addition, much materials research is supported directly by industry. The NASA program must be defined within the context of this range of ongoing activities. It is important that in defining its program, NASA continuously seek advice from a broad community of materials scientists and engineers.

The Committee recommends that NASA prepare a program document presenting the goals of the materials processing program for the next five to ten years and explaining the relationship of the goals to the activities of other programs in government and industry. Timetables and estimated costs should be included. It is important that the scientific and engineering communities be involved in meaningful ways in the formulation of the program.

The experiments that form NASA's materials processing program should be selected with great care and conducted as part of a comprehensive program that includes good earth-based experiments. Where appropriate, ground-based work should include experiments using drop towers, aircraft, or sounding rockets. Earlier in this report, specific topics were discussed from which some generalizations can be made about the areas in which convincing demonstration experiments are likely to be found.

## ORGANIZATION AND MANAGEMENT

In the near term, the NASA program will have to support extensive earth-based activities designed to supplement and provide comparative data for space experiments. In later stages, the program should envision extensive use of space facilities by non-NASA users paying for their own experiments.

The NASA program in materials should be designed to develop through two phases: (1) development and demonstration and (2) management of the Space Shuttle as a national facility. The first stage may span about the first five years of Shuttle use, but NASA should have a clear objective to move into the second phase as soon as possible, certainly before the second half of the 1980s.

### Phase One: Development and Demonstration

Phase One must be carefully planned. The only experiments flown should be those expected to clearly delineate the potentials and limitations of materials experiments in space and that also provide NASA with the experience necessary to develop facilities of maximum value to the scientific and engineering communities. Every effort should be made to involve agencies other than NASA in the planning of appropriate experimental programs. In developing a facility such as the Space Shuttle, it is important to involve potential customers as early as possible. The eventual demand for use of the Space Shuttle and Spacelab for materials processing will be determined largely by judgments reached by the materials research community about the usefulness and credibility of the results of the demonstration program.

Experimental facilities such as centrifuges, magnetic fields, buoyancy devices, drop towers, aircraft in parabolic flight, or rocket probes can be used in some experiments to alter the gravity vector. When developed, the Space Shuttle will constitute an additional facility. For some experiments, the Shuttle may prove to be simpler to use than other approaches and may permit more definitive experiments. In some cases, the Shuttle may be the only feasible way to work at low gravity. It is unlikely, however, to prove to be less expensive than alternative techniques even if only operating costs are recovered. One must expect that investigators will weigh all these considerations before deciding whether to pay for time on the Space Shuttle or use alternative facilities.

### Phase Two: The Space Shuttle as a National Facility

If the experiments carried out in Phase One convincingly demonstrate their usefulness as experimental facilities for materials processing, the Space Shuttle and Spacelab should be made available as a national resource to scientists and engineers working in universities, government laboratories, or industry on the same bases as other national experimental facilities. Individuals or groups of scientists wishing

to use the Shuttle or Spacelab should be required to pay for time on the facility. This would require convincing funding agencies and their customary peer reviewers of the value of the proposed activity. In addition, the authors of the proposals must convince the managers of the facility that their experiments are of sufficient scientific merit or technological importance to justify use of the facility and show that their experiments will conform to safety and other necessary operating requirements. As with other national research facilities, user rates should not be designed to cover the total real cost of operating the facility.

#### ADMINISTRATION

NASA management may find it difficult to ensure that the materials program moves as directly as possible over the next five to ten years toward its final objective described as Phase Two. There is a possibility that NASA could generate a large self-perpetuating program in materials, independent of and largely isolated from the many other earth-bound programs in materials processing. To avoid this, the Committee recommends that NASA establish a standing advisory panel to its materials program. In addition, NASA should enhance the credibility of its materials program with the materials research community by establishing a single, carefully organized, centrally coordinated, publicly announced peer review system for evaluating materials research proposals submitted to *any* NASA organizational element. Reviewers should be experts in the science or engineering of matters under review and not necessarily specialists in experiments in space. Except in unusual circumstances, reviewers should not themselves be recipients of current NASA grants.

To administer the program, NASA should use in-house personnel, among whom should be materials scientists and engineers who would:

- in Phase One, assess the scientific and technical merit of proposals (taking into consideration the recommendations of peer reviewers) and decide whether or not to fund the work proposed;

- in Phase Two, decide whether or not to exercise NASA's veto power;

- develop and manage NASA's research facilities in a manner responsive to an understanding of the problems and needs of scientists and engineers working in the materials field; and

- solve materials problems involved in the development of structures in space.

NASA will not be able to recruit and keep capable materials scientists and engineers if their work has a purely service function.

To increase its direct involvement in materials research, the staff managing the materials program should also be active in NASA research directed toward finding solutions to problems of engineering in space. The Committee did not attempt to assess the size of the in-house research activity necessary to directly support the total NASA mission.

The Committee believes that while there may be some justification in Phase One for including flight or ground-based experiments as a part of NASA's in-house effort, in Phase Two, the greater part of the materials processing experiments should come from outside NASA. If outside financial support is lacking, the Phase Two program should be discontinued.

## CONCLUSIONS

The objectives of this study and the related principal findings are summarized below.

*Assessment and evaluation of the scientific and technological significance of what has been learned to date about processing materials in the space environment.*

The Committee concludes that to date, the NASA program for processing materials in space has been weak. Most experiments were keyed to flight opportunities, often at the expense of carefully reasoned planning of the research. Measurement of the gravity vector prevailing during the experiments was not made with the precision needed. Scientific and technological results were sometimes shallow, incomplete, or inconclusive. Some of the work received undue publicity. Nevertheless, some work done in space has shown that valid experiments can be planned, manipulations performed, and useful samples returned to earth for study. It is noteworthy that the work that gave productive results had a sound base in terrestrial research.

*Judgment of the merit of a program on materials processing in space.*

The principal value of the space environment lies in the availability of low gravitational acceleration for long times. Opportunities for useful exploitation of this environment appear to include research on solidification, especially plane-front growth of single-phase and polyphase solids and mixed composites having phases of different density. The avoidance of containers may be facilitated in space, with benefit to the physical measurement or processing of certain reactive materials and for purification of exemplary materials. The space environment may also assist the management of aerosols in droplet processing and the control of convection in combustion studies of flame propagation and the limits of flammability.

The Committee believes, however, that instances in which a low-gravity environment is likely to be important for materials processing will be few and specific. When gravity has an adverse effect on a process, stratagems for dealing with it can usually be found on earth that are

much easier and less expensive than recourse to space flight. Experiments on processes in low gravitational fields can be expected to provide only incremental improvement upon terrestrial capabilities. Some of these specialized capabilities are summarized below in "Scientific Conclusions."

*Recommendations relating to the nature and scope of NASA's future program of experiments on materials processing in space.*

The Committee has not discovered any examples of economically justifiable processes for *producing* materials in space and recommends that this area of materials technology not be emphasized in NASA's program. The Committee has identified some activities in which experiments in space and on earth can be expected to contribute usefully to the understanding of materials processes or to the preparation of specialized exemplary materials. Research and development along these lines seems appropriate, with the initiative resting with the prospective investigators. The identification of programs for investigation must be made by peer review, not by the availability of funds or the need to use a space facility. The Committee has not tried to conclude what level of effort can be justified in such research, but believes that this activity would not involve large numbers of investigators. The definition, organization, and administration of a space program on materials were discussed earlier in this report and are summarized under "Administrative Conclusions" below.

The magnitude of gravity and the perturbations therein may be limiting criteria for some materials experiments on board the orbiting Space Shuttle. In any event, accurate knowledge of the history of the gravity vector during an experiment will be essential to interpretation of results. The Committee accordingly recommends that NASA work now to refine estimates of the gravity level that will prevail for materials experiments on the Shuttle and in Spacelab, and begin now to plan for precise measurements of gravity histories during materials experiments. If improved recording accelerometers or other instruments are required, development of such instruments should begin without delay.

## SCIENTIFIC CONCLUSIONS

A valid scientific basis exists for performing certain classes of experiments on materials in space. The environment of low gravitational acceleration for long times offers the best advantage of the space environment for such experiments. Other factors to be found in space, such as temperature, level of vacuum, or presence of high-energy radiation, can be realized better and more easily on earth.

The terrestrial science and technology of materials is generally well understood. Little expectation exists of a scientific breakthrough from appeal to a low-gravity environment. Nevertheless, reasonable

prospects can be found for useful work on materials in space, as noted above. To judge the merits of recourse to low gravity, it is essential to compare rigorously the limitations on experimentation for the same basic purpose at one g and at low g, over both short and long periods. Furthermore, the limitations imposed upon low gravity by fluctuating accelerations over long periods must be carefully assessed. Finally, critical evaluations must be made of the comparative costs and the relative likelihood of success.

Much critical analysis and professional interaction should be devoted to classes of experiments that appear to be appropriate for a low-gravity environment. The Committee believes that this environment may be useful in addressing phenomena related to fluids, including gases. Among phenomena or properties that bear on materials processing and that seem to merit study in space are

- fundamentals of convection and of coupled convective and diffusive transport;

- convection during phase changes and chemical reactions and the interactions of convective transport with transformation processes, especially those responsible for microstructures of solid materials;

- dependence of density, viscosity, thermal diffusivity, and mass diffusivity of melts and solutions on composition and temperature, particularly as influenced by buoyancy-driven convection;

- equilibrium properties and dynamic phenomena at gas-melt and melt-melt interfaces, beginning with surface tensions and interfacial tensions as functions of temperature, composition, absorption of soluble trace contaminants, or accumulation of meniscus-seeking insoluble contaminants;

- phenomena associated with the intersections of fronts and menisci with solid walls (for example, edge effects in solidification and combustion) and contact angles, wetting, melt spreading, and junctions where gas, liquid, and solid meet in three-phase contact lines;

- tests of theoretical models of fluid flow systems that experience complicated combinations and distributions of forces or have complex compositions; and

- parameters related to instabilities associated with critical phenomena.

The Committee sees need to emphasize two points that emerged from the testimony of its advisors and from the experience of materials study in space to date. First, the space environment usually contributes at

least as many problems as it solves. In sophistication, reliability, convenience, and cost, terrestrial experimentation is generally superior to what can be expected in space. Second, space experimentation will have little value unless its planning is founded on substantial earth-based information and unless the results are coupled to those of complementary terrestrial programs.

#### ADMINISTRATIVE CONCLUSIONS

Federal agencies and industry have responsibility to support the materials science and technology germane to their missions; it is essential that the NASA program on materials be defined within the context of this range of activities. In developing its program, NASA must continuously seek advice from a broad community of materials scientists and engineers. High levels of expertise are required, as are unbiased judgments. The best means available are the normal peer review system and independent advisory groups. Possible users of the results of the program should be closely involved in its formulation. NASA must keep in mind that one of its proper roles is the development and operation of singular facilities in response to and not independent of the scientific and technological community.

The final test of the merit of a space program in materials science and technology must be the user community's judgment on priorities for the resources available. In development of any program, NASA must depend on this consensus. NASA should continue to develop its technical capabilities in space and make them known to the materials community. NASA should not, however, presume to take an independent role in the establishment of programs and facilities for materials science and engineering.

With support from the materials community, NASA should prepare a program plan for the next five years describing specific materials activities and relationships with other programs supported by government and industry and providing budgets and timetables. The materials science and materials engineering communities should be closely involved in the formulation of the program. The experimental parts of this program should be chosen with great care from earth-based scientific and technological work that clearly indicates that recourse to the space environment might advance the results.

The merit of a program for materials processing in space will ultimately be tested by the participation of investigators whose support comes from industry or from federal funding agencies other than NASA. Space facilities, including the Space Shuttle and Spacelab, if they are viable for materials science and technology, will emerge as national facilities comparable to what is available today from the national centers for astronomy, magnetism, or high energy physics. The Committee visualizes two stages in the NASA program on materials: first, development and demonstration, and second, management of national facilities.

The first stage might occupy a span of about five years, would require careful planning, and would be limited to studies expected to delineate clearly the potentials and limitations of materials experimentation in space. During the second stage, NASA may well have to continue to employ a small number of materials scientists and engineers to adequately fulfill the need for competent program management. These scientists and engineers might usefully be employed also in the development of new materials for use in the space program.

In summary, the Committee concludes that prospects for using the space environment for science and technology related to materials processing take the form of incremental advantages over earth-based processes, rather than breakthroughs into new science and technology.

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## APPENDIX

### MATERIALS PROCESSING EXPERIMENTS TO DATE

Materials processing experiments performed aboard Apollo, Skylab, Apollo-Soyuz Test Project, and Space Processing Applications Rocket (SPAR) Project flights are listed in this appendix. The title shown for each experiment is that used by NASA in its agreement with the principal investigator(s) and the numerical designation is that assigned in the NASA experiment-numbering system. The flight(s) during which the experiment was performed and the principal investigator(s) are noted, followed by a brief description of the experiment or its objective and the principal conclusions.

References are provided for the reader who may wish to know more about the experiment. It should be noted that the title of a report may differ from the title of the experiment and that the authors of the report may be other than the principal investigators. If the results of the experiment have been reported in a scientific or technical journal, additional references may be given.

The conclusions are taken from NASA reports on the results of the experiments. The Committee has not examined the validity of the conclusions. In some cases, objectives and conclusions have been taken verbatim from the cited references; others are paraphrased.

It should be noted that almost one-half of the conclusions cited are taken from summary reports published less than one year after the experiments were performed. Some experimenters have reached new conclusions, based on further study of their data, or modified their earlier conclusions and have reports in preparation presenting these later results. The interested reader should search the literature or contact the experimenter directly.

For convenience, the experiments have been grouped into the following categories:

- Crystal Growth
- Solidification (Skylab, Apollo, and Apollo-Soyuz Flights)
- Solidification (SPAR Flights)

Fluid Dynamics  
Combustion  
Electrophoresis

At the time this report went to press, a few more sounding rocket experiments had been performed but their results not yet published. These are listed at the end of the appendix so that those interested may seek the results later.

## CRYSTAL GROWTH

### Microsegregation in Germanium

M 559 Skylab 3 September 1973

J. T. Yue and F. A. Padovani

To determine if an improvement over control crystals grown on the ground could be obtained in solute microsegregation for crystal growth of semiconductor material in a gravity-free environment.

Reported Conclusion: Solidification of doped germanium in space can provide six-fold improvement in macrosegregation and nearly two-fold improvement in microsegregation for crystal growth by the gradient freeze method as compared to earth-grown control crystals.

Reference: "Influence of Gravity-free Solidification on Microsegregation," John T. Yue and Fred W. Voltmer. Proceedings, Third Space Processing Symposium: Skylab Results, Vol. 1, NASA M-74-5, George C. Marshall Space Flight Center, June 1974, pp. 375-424. See also "Influence of Gravity-free Solidification on Solute Microsegregation," J. T. Yue and F. W. Voltmer. Journal of Crystal Growth 29:329-341 (1975).

### Growth of Spherical Crystals

M 560 Skylab 3 September 1973; Skylab 4 December 1973

H. U. Walter

To investigate the feasibility of containerless processing of single crystals in the space environment; to obtain information on the structural perfection of space-grown crystals as compared to samples grown on earth; to demonstrate the potential of space for producing homogeneously doped semiconductor material.

Reported Conclusions: Highly perfect single crystals can be prepared by seeded containerless solidification. Dopant inhomogeneities were observed, although all indications point to essentially no-fluid-flow conditions. Production of homogeneously doped single crystals by containerless techniques appears to be feasible.

Reference: "Seeded, Containerless Solidification of Indium Antimonide," H. U. Walter. Skylab Results, pp. 257-273. See also "A Mechanism for Generation of Pulsating Growth and Nonrotational Striations during Initial Transient Solidification," H. U. Walter. Journal of the Electrochemical Society 123:1098-1105 (1976) and "Generation and Propagation of Defects in Indium Antimonide," H. U. Walter. Journal of the Electrochemical Society 124:250-258 (1977).

#### Indium Antimonide Crystals

M 562 Skylab 3 September 1973; Skylab 4 December 1973  
A. F. Witt and H. C. Gatos

To confirm the advantages of the zero-gravity environment; to obtain basic data on solidification; to explore the feasibility of electronic materials processing in space.

Reported Conclusion: The experiment proved unambiguously the uniqueness of zero-gravity conditions for directly obtaining fundamental data in crystal growth and segregation associated with solidification.

Reference: "Steady State Growth and Segregation under Zero Gravity: InSb," A. F. Witt et al. Skylab Results, pp. 275-299. See also "Crystal Growth and Steady-State Segregation under Zero Gravity: InSb," A. F. Witt et al. Journal of the Electrochemical Society 122(2):276-283 (1975).

#### Interface Marking in Crystals

MA 060 Apollo-Soyuz July 1975  
H. C. Gatos and A. F. Witt

To establish under near-zero-gravity conditions the absence or presence of convection phenomena, the surface tension of the melt, microscopic growth-rate behavior during directional solidification, the dopant segregation behavior and its dependence on the microscopic growth rate, and the heat transfer characteristics of the solidification system.

Reported Conclusion: The experiment revealed growth and segregation effects previously not observed on earth that could not be accounted for by existing experimental or theoretical models.

Reference: "Quantitative Determination of Zero-Gravity Effects on Electronic Materials Processing: Germanium Crystal Growth with Simultaneous Interface Demarcation," H. C. Gatos et al. Apollo-Soyuz Test Project: Composite of MSFC Final Science Report (ASTP Final Report), NASA Technical Memorandum TMX-73360, George C. Marshall Space Flight Center, January 1977, pp. V-1 to V-65.

#### Vapor Growth of IV-VI Compounds

M 556 Skylab 3 September 1973; Skylab 4 December 1973

H. Wiedemeier

To observe and measure changes in the mass transport rate of a chemical system and in the morphology of crystals of IV-VI compounds.

Reported Conclusions: The experimental evidence confirmed the predicted positive effects of microgravity on crystal quality. Mass transport rates observed were greater than expected in the microgravity environment.

Reference: "Vapor Growth of GeSe and GeTe Single Crystals in Microgravity," H. Wiedemeier et al. Skylab Results, pp. 235-256. See also "Crystal Growth and Transport Rates of GeSe and GeTe in Microgravity Environment," H. Wiedemeier et al. Journal of Crystal Growth 31:36-43 (1975).

#### Mixed III-V Crystal Growth

M 563 Skylab 3 September 1973; Skylab 4 December 1973

W. R. Wilcox and R. A. Lefever

To test whether grains are generated by the compositional variations arising from hydrodynamic fluctuations in the melt.

Reported Conclusions: Concentration profiles and compositional homogeneity were strongly influenced by the magnitude and direction of gravity. Lack of convective stirring in space processing led to initial compositional transients. A wide variety of grain sizes was observed but with no trend yet observed. There appears to be no large advantage to space processing of alloys from this standpoint.

Reference: "Directional Solidification of InSb-GaSb Alloys," James F. Yee et al. Skylab Results, pp. 301-374. See also "Influence of Gravity on Crystal Defect Formation in Indium Antimonide-Gallium Antimonide Alloys," J. F. Yee et al. Journal of Crystal Growth 30(2):185-192 (1975).

#### Crystal Growth in Space

MA 028 Apollo-Soyuz July 1975

M. David Lind

To investigate the growth of single crystals of insoluble substances by a process in which two or more reactant solutions are allowed to diffuse toward each other through a region of pure solvent.

Reported Conclusion: The experiment successfully proved the feasibility of a novel method of crystal growth, both for producing superior crystals

of a variety of compounds and for promoting a better understanding of the theory of crystal growth.

Reference: "Crystal Growth: Experiment MA 028," M. David Lind. Apollo-Soyuz Test Project Preliminary Science Report, NASA SP 412, Lyndon B. Johnson Space Center, Houston, Texas, February 1977, pp. 525-582.

#### Crystal Growth from the Vapor Phase

MA 085 Apollo-Soyuz July 1975

H. Wiedemeier

To extend and generalize microgravity crystal growth for a class of compounds and to characterize the unexpected gas motion observed on prior Skylab experiments.

Reported Conclusions: The results demonstrated a considerable improvement in structural and chemical homogeneity, surface morphology, and bulk perfection of space-grown materials compared to ground-based specimens. Mass transport rates greater than those predicted by past models were observed. There was excellent agreement between the results of the ASTP experiment and those of Skylab (experiment M 556).

Reference: "Crystal Growth from the Vapor Phase," H. Wiedemeier et al. ASTP Final Report, pp. VII-1 to VII-40. See also "Morphology and Transport Rates of Mixed IV-VI Compounds in Microgravity," H. Wiedemeier et al. Journal of the Electrochemical Society 124:1095-1102 (1977).

#### Multiple Materials Melting

MA 150 Apollo-Soyuz July 1975

V. S. Zemskov, V. N. Kubasov, I. N. Belokurova, A. N. Titkov,

I. L. Shulpina, V. I. Safarov, and N. B. Guseva

To study the possibility of using zero-g conditions for obtaining solid solution monocrystals with uniformly distributed components.

Reported Conclusions: Under zero-g conditions, monocrystals of Sb-doped GeSi solid solution were obtained with and without seeding by means of directional crystallization. During melting and crystallization under zero-g conditions, convective mixing was either absent or so negligible that it did not affect the process.

Reference: "Germanium-Silicon Solid Solutions," V. S. Zemskov et al. ASTP Final Report, pp. IX-1 to IX-36. See also "Proceedings (Doklady) of the Academy of Sciences of the USSR," Vol. 233, March 11, 1977, pp. 341-344 (in Russian).

## SOLIDIFICATION (SKYLAB, APOLLO, AND APOLLO-SOYUZ FLIGHTS)

### Composite Casting

Apollo 14 January 1971

I. C. Yates, Jr., J. L. Reger, W. H. Steurer, and R. Fabiniak

To obtain preliminary data on the processes of melting, mixing, and solidification of composite materials in space.

Reported Conclusions: Low-g composite casting demonstrations served their purpose in that material structures were produced that cannot be duplicated on earth. Where dispersants such as fibers, particles, or gases had been added to the matrix, enhanced dispersion and distribution were found in the space-processed samples. Normally immiscible mixtures showed stable dispersions unattainable on earth.

Reference: Apollo 14 Composite Casting Demonstration Final Report, I. C. Yates, Jr. NASA Technical Memorandum TMX-64641, George C. Marshall Space Flight Center, October 1971.

### Metals Melting

M 551 Skylab 2 June 1973

R. M. Poorman and E. C. McKannan

To study the behavior of molten metal in low gravity with particular attention to the stability of the molten puddle and its interface with the solidified metal; to characterize metals solidified in low gravity with regard to grain size, orientation, and subgrain patterns; to determine the feasibility of joining and casting metals in space.

Reported Conclusions: It is feasible to do electron beam welding, cutting, and melting in the low-gravity environment of space. Grain size in the space specimens was smaller than had been observed on earth. The grain growth was more equiaxed in space than on earth.

Reference: "Skylab M 551 Metals Melting Experiment," E. C. McKannan and R. M. Poorman. Skylab Results, pp. 85-100.

### Exothermic Brazing

M 552 Skylab 2 June 1973

J. R. Williams and C. M. Adams

To evaluate brazing as a tube-joining technique for the assembly and repair of hardware in space and to study the spreading, mixing, and capillary action of molten braze material in near-zero gravity.

Reported Conclusions: The absence of gravity greatly extends the usefulness of brazing. The surface tension forces driving capillary flow

predominate in a zero-gravity environment. Liquid-vapor boundary surfaces (menisci) and the flow of liquid metal driven by surface tension are in close conformance with what had been predicted for zero-gravity environment. The presence or absence of gravity has no observable effect on the mechanism of alloy solidification. Such microstructural details as dendritic configuration and eutectic structure were the same in space as on earth. The Skylab specimens exhibited fewer and smaller shrinkage defects than the comparative ground-processed characterization samples, indicating that gravity forces are significant during the capillary movement of the braze alloy. The oxide build-up on both the Skylab braze alloy and the substrate materials was less than on ground-based specimens, indicating the adequacy of utilizing the space vacuum and its infinite pumping capacity for brazing operations of this type.

Reference: "Skylab Experiment M 552 Exothermic Brazing," J. R. Williams. Skylab Results, pp. 33-84.

#### Sphere Forming

M 553 Skylab 2 June 1973

E. A. Hasemeyer and D. J. Larson, Jr.

To study the effects of weightlessness in containerless solidification processes of four face-centered cubic materials.

Reported Conclusions: Typically, the one-g specimens had a sphericity ( $R_{\max}/R_{\min}$ ) of 1.28, whereas the flight samples were typically 1.01 to 1.04, a substantial enhancement due to the reduction in gravity. The record of terracing was excellent although not unprecedented in earth-based work. Although thermal control has previously been considered important, pressure control may prove to be an important consideration as well. As the pressure head is released (in low-g), low pressure phase reactions may occur in the bulk that could not occur otherwise. These reactions are sufficiently rare terrestrially as to be unnamed.

Reference: "Skylab M 553 Sphere Forming Experiment," D. J. Larson, Jr. Skylab Results, pp. 101-113.

#### Silver Grids Melted in Space

M 565 Skylab 3 September 1973

A. Deruyttere

To make a preliminary study of the behavior of porous material when melted and resolidified in weightless condition.

Reported Conclusions (Provisional): Most of the original porosity in the samples disappeared during the melting stage. The shape and surface condition of a sample melted and solidified in space were not determined only by surface tension. Leveling-out of concentration gradients appeared to be slow in the molten metal when gravity-induced convection was absent.

Reference: "Silver Samples Melted in Space, Skylab Experiment M 565," A. Deruyttere et al. Skylab Results, pp. 159-202.

#### Immiscible Alloy Compositions

M 557 Skylab 3 September 1973; Skylab 4 December 1973

J. L. Reger

To study potentially useful immiscible alloys using the M 512/518 Multipurpose Electric Furnace.

Reported Conclusions: Low-gravity processed specimens exhibited homogenization and microstructural appearances better than the one-g control specimens. Low-gravity processing of materials having liquid or solid immiscibility can provide compositions exhibiting unusual metallographic and electronic behavior.

Reference: "Experiment No. M 557, Immiscible Alloy Compositions," J. L. Reger. Skylab Results, pp. 133-158.

#### Alkali Halide Eutectics

M 564 Skylab 3 September 1973

A. S. Yue

To prepare fiber-like NaCl-NaF eutectics with continuous NaF fibers embedded in a NaCl matrix; to examine the eutectic microstructure; to measure the relevant optical properties of space-grown and earth-grown eutectics.

Reported Conclusions: Continuous NaF fibers were produced in the Skylab experiments. Success in producing continuous fibers was due to the absence of convection current in the liquid during solidification. Larger transmittance over a wider wavelength was obtained from the Skylab-grown ingots.

Reference: "Halide Eutectic Growth," A. S. Yue and J. G. Yu. Skylab Results, pp. 469-489.

#### Whisker-Reinforced Composites

M 561 Skylab 3 September 1973; Skylab 4 December 1973

Tomoyoski Kawada

To obtain Ag and SiC whisker composites with high density and uniform distribution of whiskers.

Reported Conclusions: In both the Skylab and ground-based samples, the density ratio varied when the sample was melted and pressurized. Variations in distribution density of whiskers occurred in ground-based test samples but not in Skylab samples. The microhardness of earth-based

samples was smaller than for the Skylab samples and showed large fluctuations along the axial direction as compared to the Skylab samples.

Reference: "Preparation of Silicon Carbide Whisker Reinforced Silver Composite Material in a Weightless Environment, Skylab Experiment M 561," Tomoyoski Kawada et al. Skylab Results, pp. 203-233.

#### Copper-Aluminum Eutectic

M 566 Skylab 3 September 1973; Skylab 4 December 1973

Earl A. Hasemeyer

To determine if an improved structure could be grown in the absence of gravity-induced thermal convection.

Reported Conclusion: Specimens processed in zero gravity were superior to ground-based specimens with respect to defect spacing in lamellar widths by 12% and in fault density by 20%.

Reference: "Skylab Experiment M 566 Copper-Aluminum Eutectic," E. A. Hasemeyer et al. Skylab Results, pp. 457-467.

#### Monotectic and Syntectic Alloys

MA 044 Apollo-Soyuz July 1975

C. Y. Ang

To investigate the effects of weightlessness on the melting and solidification of two material systems, lead-zinc and aluminum antimonide.

Reported Conclusions: Liquid-state homogenization of polycrystalline multi-phase AlSb at low gravity produced major improvements in macroscopic and microscopic homogeneity. The experiment with Pb-Zn suggested that there is significant inaccuracy in the published phase diagram for Pb-Zn.

Reference: "Monotectic and Syntectic Alloys," L. L. Lacy and C. Y. Ang. ASTP Final Report, pp. IV-1 to IV-51.

#### Zero-G Processing of Magnets

MA 070 Apollo-Soyuz July 1975

D. J. Larson, Jr.

To test the effect of the reduction of gravitationally dependent elemental segregation and convection on high coercive strength magnetic composites.

Reported Conclusions: Fluid static configurations in low gravity were appreciably different from those in one g but were found to agree well with theory. Bismuth undergoes a liquid phase transition with large

hysteresis at 988°K on heating and 858°K on cooling. The intrinsic coercive strength of as-grown low-g MnBi/Bi eutectic samples greatly exceeds any value previously reported for this magnetic composite. The solidification product from the orbital processing of the Bi/MnBi faceted rod eutectic differs significantly in particle numbers and size distribution from equivalently processed terrestrial samples.

Reference: "Zero-G Processing of Magnets: Experiment MA 070," D. J. Larson, Jr. ASTP Final Report, pp. VI-1 to VI-53.

#### Sodium Chloride-Lithium Fluoride Eutectic

MA 131 Apollo-Soyuz July 1975

A. S. Yue

To prepare fiber-like LiF-NaCl eutectic with continuous LiF fibers embedded in the NaCl matrix and to make an analysis of the material.

Reported Conclusions: Continuous LiF fibers regularly arranged in a portion of the NaCl matrix that had been resolidified unidirectionally in a space environment were produced. Larger transmittance over a wider wavelength and better image transmission were obtained for transverse sections of the ASTP-grown ingots.

Reference: "Zero Gravity Growth of NaCl-LiF Eutectic Experiment MA 131," A. S. Yue et al. ASTP Final Report, pp. VIII-1 to VIII-27.

### SOLIDIFICATION (SPAR FLIGHTS)

#### Lead-Antimony Eutectic

74-5 SPAR-I December 1975; SPAR-II May 1976

Robert B. Pond and J. W. Winter, Jr.

To ascertain whether it is possible to get a faithful and complete eutectic structure in 88.8 Pb-11.2 Sb in microgravity.

Reported Conclusions: The dual primary crystallization products are the result of thermal supercooling in the Pb-Sb alloys of SPAR-I and SPAR-II. The microgravity field experienced during solidification of the SPAR-I and SPAR-II specimens caused the crystallization products to be homogeneously dispersed. All other gravity fields studied (1 g, 25 g, 280 g and 1000 g) produced more erratic dispersion of the Pb dendrites.

Reference: "Space Solidification of Pb-Sb Eutectic: Experiment 74-5," Robert Pond et al. Space Processing Applications Rocket Project: SPAR-II Final Report, TMX-78125, George C. Marshall Space Flight Center, November 1977, pp. III-1 to III-29. See also same title, Robert B. Pond et al. Space Processing Applications Rocket Project: SPAR-I Final

Report, NASA TMX-3458, George C. Marshall Space Flight Center, December 1976, pp. I-1 to I-31.

Foams from Sputter-Deposited Metals

74-10 SPAR-I December 1975; SPAR-II May 1976

J. W. Patten and E. N. Greenwell

To produce metal foam materials from sputtered metal deposits.

Reported Conclusions: (SPAR-I) Metal foam materials were produced in a zero-gravity environment. Data were obtained that, in combination with data from a repetition on SPAR-II, were expected to reveal some effects of gravity on formation of metal foams as compared with metal foams produced on earth. The data indicated, in a preliminary way, the potential for variation in foam structure. (SPAR-II) In the thickest samples foamed in zero-gravity, more bubble coarsening and a larger void volume fraction were observed with increasing time above the melting point. Effects of oxide scale were pronounced and inhibited obtaining kinetic information on foam formation.

Reference: "Feasibility of Producing Closed-Cell Metal Foams in a Zero-Gravity Environment From Sputter-Deposited Inert Gas-Bearing Metals and Alloys: Experiment 74-10," J. W. Patten and E. N. Greenwell. SPAR-I Final Report, pp. II-1 to II-62. See also same title, J. W. Patten and E. N. Greenwell. SPAR-II Final Report, pp. IV-1 to IV-46.

Particle-Interface Interactions

74-15 SPAR-I December 1975

Donald R. Uhlmann

To study the interaction of second-phase particles with a solidification front.

Reported Conclusion: The principal value of the experiment was the insight it provided -- not directly into the behavior of second-phase particles at a solidification front, but into materials and environmental factors important in elucidating the phenomena from rocket experiments. (Repeated on SPAR-IV, June 1977, results not yet reported.)

Reference: "Uniform Dispersions of Crystallization Processing: Experiment 74-15," Donald R. Uhlmann. SPAR-I Final Report, pp. III-1 to III-40.

Dendrite Remelting and Macrosegregation

74-21 SPAR-I December 1975; SPAR-II May 1976

M. H. Johnston and C. S. Griner

To observe the growth of dendrites in the columnar solidification region

in order to determine the influence of gravity-driven flow on the formation of the equiaxed zone.

Reported Conclusions: (SPAR-I) When  $\text{NH}_4\text{Cl}$  was solidified in low gravity, only four nuclei grew to form the complete casting. There were no free-flowing crystals or visible dendrite remelting. The lack of fluid flow allowed symmetrical dendrite growth into the fluid. Some necking of secondary arms occurred, but no coarsening or fragmentation resulted. The growth rate of the interfaces was less than that of individual dendrites. Total growth was columnar with no equiaxed zone being formed. (SPAR-II) The absence of sample solidification during the period of low-gravity flight precluded achieving the experiment objectives.

Reference: "The Direct Observation of Solidification as a Function of Gravity Levels: Experiment 74-21," M. H. Johnston and C. S. Griner. SPAR-I Final Report, pp. V-1 to V-19; "The Direct Observation of Dendrite Remelting and Macrosegregation: Experiment 74-21," M. H. Johnston and C. S. Griner. SPAR-II Final Report, pp. V-1 to V-13. See also Metallurgical Transactions 8(A):77-82 (1977).

#### Thoria Dispersed Magnesium

74-34 SPAR-I December 1975; SPAR-II May 1976

Louis Raymond and Choh-Yi Ang

To demonstrate achievement of optimum distribution of dispersed thoria particles in an Mg matrix upon melting and solidification in short duration at a low-gravity level.

Reported Conclusions: (SPAR-I) Low-gravity effects contributed to the soundness of the casting. Optimum distribution of dispersoids was not achieved. (SPAR-II) Utilizing the gettering action of Th metal in the Th-MgO-Mg melt, low-gravity effects contributed to significantly greater uniformity in the dispersion of heavy thoria particles and greater hardness than the earth-processed counterparts. Soundness of the low-gravity castings was also observed.

Reference: "Casting Dispersion-Strengthened Composites at Zero Gravity: Experiment 74-34," Louis Raymond and C. Y. Ang. SPAR-I Final Report, pp. VI-1 to VI-34. See also same title, L. Raymond and C. Y. Ang. SPAR-II Final Report, pp. VII-1 to VII-51.

#### Contained Polycrystalline Solidification

74-37 SPAR-I December 1975

John M. Papazian and Theodoulos Z. Kattamis

To investigate the effect of low gravity on the width of a solute enriched zone in polycrystalline metallic solidification.

Reported Conclusions: A darker green layer observed ahead of the solid-liquid interface was most likely the solute enriched zone and appeared to become wider in the flight specimen. The irregular shape of the interface in the flight specimen, the smaller grain size, the equiaxed grain morphology, and the larger average macroscopic growth rate were attributed to parasitic nucleation ahead of the solid-liquid interface. (Additional experiment on SPAR-IV, June 1977, results not yet reported.)

Reference: "Contained Polycrystalline Solidification in Low Gravity: Experiment 74-37," J. M. Papazian and T. Z. Kattamis. SPAR-I Final Report, pp. VIII-1 to VIII-21.

#### Aluminum-Indium Alloys

74-62 SPAR-II May 1976

H. Ahlborn and K. Lohberg

To determine if alloys of different compositions exhibit different structures depending on the separation mechanism.

Reported Conclusions: The flight samples did not provide any reliable information about the decomposition of the homogeneous melt into two immiscible melts. Unexpected information was obtained about the process of separation of the two phases into an Al-rich region and an In-rich region not observed with samples treated the same way on earth. A number of questions have been raised that require further experiments.

Reference: "Segregation and Solidification of Liquid Aluminum-Indium Alloys Under Zero Gravity Conditions," Karl Lohberg et al. SPAR-II Final Report, pp. VIII-1 to VIII-44.

#### Dispersion Strengthened Pb-Ag Alloys

74-63 SPAR-I December 1975

Werner Heye

To produce a second order or third order superconductor in the form of a mixture of a first order superconductor (lead) and a normal electrical material (silver).

Reported Conclusion: A transition of a first order superconductor to a second order superconductor in the form of a mixed state took place in the flight sample only.

Reference: "Preparation of a Special Alloy Under Zero-Gravity for Magnetic Hard Superconductors: Experiment 74-63," W. Heye and M. Klemm. SPAR-I Final Report, pp. IX-1 to IX-22.

### Agglomeration in Immiscible Liquids

74-30 SPAR-II May 1976

S. H. Gelles and A. J. Markworth

To gain an understanding of the influence of gravity, cooling rate, and composition on the structure of liquid-phase immiscible systems. To determine the effect of gravity on the structure of two aluminum alloys when cooled through the miscibility gap at a controlled rate.

Reported Conclusions: An unexpected type of macrostructure resulted from processing the Al-40 weight percent In and Al-70 weight percent In samples in space. The morphological evolution has been interpreted in terms of fluid flow occurring in the low-gravity environment. Fluid flow at low gravity can arise from numerous sources. Of the sources analyzed, thermocapillary convection and conventional convection are probably active. Capillary flow has as yet not been analyzed but probably is important. Residual fluid motion due to rocket spin does not appear to make an important contribution. The equilibrium configuration of Al and In in a low-g environment has been calculated on the basis of known surface energies of the components and assumed values of the interfacial energy based on those of similar systems. A configuration consisting of an annular ring of In surrounding an Al-rich core is predicted and agrees closely with the observations in the present system as well as with some past results.

Reference: "Agglomeration in Immiscible Liquids: Experiment 74-30," S. H. Gelles and A. J. Markworth. SPAR-II Final Report, pp. VI-1 to VI-53.

## FLUID DYNAMICS

### Heat Flow and Convection

Apollo 14 January 1971

T. C. Bannister, B. R. Facemire, and P. G. Grodzka

To demonstrate the combined effect of various forces on the kind and magnitude of fluid flows that occur in actual flight.

Reported Conclusions: The flow pattern experiment confirmed conclusively the theoretical prediction that surface tension alone can cause cellular convection. Contained fluids under nominally zero-g environments can sustain steeper temperature gradients than they can under one-g conditions. Therefore, manufacturing processes that depend on carefully controlled thermal environments could be more easily accomplished in space. In any contemplated process in which a free or uncontained liquid is subjected to a temperature or concentration gradient, sizable convection can be assumed under Apollo 14 environmental conditions.

Reference: Heat Flow and Convection Demonstration (Apollo 14), T. C. Bannister. NASA Technical Memorandum TMX-64735, George C. Marshall Space Flight Center, March 1973. See also "Heat Flow and Convection Demonstration Experiments Aboard Apollo 14," P. G. Grodzka and T. C. Bannister. Science 176:506-508 (May 1972).

#### Heat Flow and Convection

Apollo 17 December 1972

T. C. Bannister, B. R. Facemire, P. G. Grodzka, L. W. Spradley, S. V. Bourgeois, and R. O. Hedden

To demonstrate the combined effects of various forces on the kind and magnitude of fluid flows that occur in actual flight using equipment of improved design over that used in the Apollo 14 flight.

Reported Conclusions: The size of the observed surface-tension-driven convection cells agreed fairly well with those predicted by linear analysis of surface-tension-driven, cellular convection. Convection occurred at lower temperature gradients in low gravity than in one g. Surface tension and gravity, therefore, apparently do not reinforce each other in a manner predicted by one analysis of cellular convection. No significant convection was observed in the radial or lineal heating experiments. The data, however, validate the accuracy of the measuring technique and allow the conclusion that the convection observed in the Apollo 14 radial and zone cells was probably caused by the experimental apparatus and spacecraft vibrations.

Reference: Apollo 17 Heat Flow and Convection Experiments Final Data Analyses Results, T. C. Bannister et al. NASA Technical Memorandum TMX-64772, George C. Marshall Space Flight Center, July 1973. See also "Heat Flow and Convection Experiments Aboard Apollo 17," P. G. Grodzka and T. C. Bannister. Science 187:165-167 (January 1975).

#### Radioactive Tracer Diffusion

M 558 Skylab 3 September 1973

A. O. Ukanwa

To determine the self-diffusion coefficients for liquid zinc in a convection-free environment and to estimate the reduction in convective mixing in space as compared to on earth.

Reported Conclusions: Radioactive isotopes can be successfully used for experiments in space to study liquid metal diffusion. Complications arising from convection in liquids during mass transfer on earth may be avoided or minimized by utilizing a zero-g environment.

Reference: "Radioactive Tracer Diffusion," A. O. Ukanwa. Skylab Results, pp. 425-456.

### Surface Tension Induced Convection

MA 041 Apollo-Soyuz July 1975

R. E. Reed, W. Uelhoff, and H. L. Adair

To detect possible convection caused by a steplike compositional variation in a liquid metal in a microgravity environment.

Reported Conclusions: Convective effects were observed but were not sufficiently large to bring about total mixing in the space flight specimens. Convection effects were large enough to prohibit analysis of the pure diffusion process. Results for the ground-based specimens were totally different from those melted in space, with convection being a dominant factor.

Reference: "Surface Tension Induced Convection in Encapsulated Liquid Metals in a Microgravity Environment," R. E. Reed et al. ASTP Final Report, pp. III-1 to III-87. See also Surface Tension Induced Convection in Encapsulated Liquid Metals in Microgravity: Apollo-Soyuz Test Project Experiment No. MA-041, R. E. Reed et al. ORNL-TM-5480, Oak Ridge National Laboratory, December 1976.

### Liquid Mixing

74-18 SPAR-I December 1975

Charles F. Schafer

To illustrate the nature of the space processing sounding rocket accelerational environment by its effects on a confined fluid system containing density gradients.

Reported Conclusions: Residual accelerations aboard the rocket were very low, so the SPAR experiment package provided a good platform for experiments requiring up to 5 minutes of low-g time. Even at very low-g levels, convective fluid motion can occur. (Additional experiment on SPAR-III, December 1976, results not yet reported.)

Reference: "Liquid Mixing Experiment: Experiment 74-18," Charles F. Schafer. SPAR-I Final Report, pp. IV-1 to IV-37.

### Bubble Behavior in Melts

74-36 SPAR-I December 1975

J. M. Papazian and W. R. Wilcox

To observe directly the interaction of solidification interfaces with bubbles and to observe the migration of bubbles in a temperature gradient in a liquid in the absence of gravitational forces.

Reported Conclusions: The effect of gravity on the grown-in void content in the CBr<sub>4</sub> specimens illustrated the potential problem posed by

bubble generation during solidification in the orbital environment. In low gravity, bubble nucleation and growth also occurred but the bubbles did not detach from the interface and the dendritic growth front was able to go around the bubbles, thus forming a void. (Additional experiment on SPAR-III, December 1976, results not yet reported.)

Reference: "Thermal Migration of Bubbles and Their Interaction with Solidification Interfaces: Experiment 74-36," J. M. Papazian and W. R. Wilcox. SPAR-I Final Report, pp. VII-1 to VII-30.

## COMBUSTION

### Zero Gravity Flammability

M 479 Skylab 4 February 1974  
J. H. Kimzey

A manned space flight engineering experiment to provide information on flammability of materials for manned spacecraft. Tests were made using six materials.

Reported Conclusions: Ignition in orbital flight was the same as at one g. Burning rates were slower than in one g. One-g testing for flammability provides an adequate test for fire safety.

Reference: "Zero Gravity Flammability," J. H. Kimzey. Skylab Results, pp. 115-130.

## ELECTROPHORESIS

### Electrophoretic Separation

Apollo 14 January 1971

E. C. McKannan, A. C. Krupnick, R. N. Griffin, and L. R. McCreight

To demonstrate the principle and problems of zone electrophoresis in space using model materials.

Reported Conclusions: Electrical and fluid flow systems of the apparatus worked as designed; gas bubbles were filtered and absorbed even in near-zero gravity. In a red-blue dye separation, resolution in space was better than on earth. The shape and sharpness of the advancing boundary of separated material were improved in space by lack of sedimentation and convection currents suppressed by the near-zero-gravity condition.

Reference: Electrophoresis Separation in Space: Apollo 14, E. C. McKannan et al. NASA Technical Memorandum TMX-64611, George C. Marshall Space Flight Center, August 1971.

### Electrophoretic Separation

Apollo 16 April 1972

R. S. Snyder, A. C. Krupnick, R. N. Griffin, and L. R. McCreight

An experiment using an improved version of the Apollo 14 apparatus to separate by electrophoresis large polystyrene latex particles as a model for the separation of biological particles in later flight experiments.

Reported Conclusions: Electrophoresis of model particles in a free liquid in a weightless environment was demonstrated. Problems arose because of electroosmosis in the absence of which a distinct separation of the two sizes of polystyrene latex particles would have been obtained.

Reference: Electrophoresis Demonstration on Apollo 16, R. S. Snyder. NASA Technical Memorandum TMX-64724, George C. Marshall Space Flight Center, November 1972. See also "Free Fluid Particle Electrophoresis on Apollo 16," R. S. Snyder et al. Separation and Purification Methods 2(2):259-282 (1973).

### Electrophoresis Technology

MA 011 Apollo-Soyuz July 1975

R. E. Allen, R. E. Bigazzi, G. H. Barlow, and Milan Bier

To separate fixed red blood cells (rabbit, human and horse), lymphocytes, and kidney cells by electrophoresis.

Reported Conclusions: Electroosmosis as a major obstacle to electrophoretic separations in closed tubes was eliminated; an enrichment of urokinase-producing cells occurred in the separation of kidney cells.

Reference: "Column Electrophoresis on the Apollo-Soyuz Test Project," R. E. Allen et al. ASTP Final Report, pp. I-1 to I-68.

### Electrophoresis Experiment

MA 014 Apollo-Soyuz July 1975

K. H. Hannig

To investigate and evaluate the increase in sample flow-rate and resolution achievable in space.

Reported Conclusions: The feasibility of free-flow electrophoresis for future experiments in space was confirmed. Increased separation of the chamber walls, permitted because convective disturbances owing to joule heating are absent in low g, can provide up to ten-fold increase in throughput. In a separation chamber of large cross section, temperature conditions required for biological materials could be met and the possibility of separating living cells under zero-g conditions was demonstrated.

Reference: "Electrophoresis Experiment MA 014," K. H. Hannig et al.  
ASTP Final Report, pp. II-1 to II-38.

ADDITIONAL EXPERIMENTS FOR  
WHICH RESULTS ARE NOT YET PUBLISHED

Gallium Arsenide and Garnet Epitaxy

74-45 SPAR-III December 1976

M. D. Lind

Beryllium Grain Refinement

74-48 SPAR-III December 1976

G. J. London and G. Wouch

Viscous Coalescence

74-53 SPAR-III December 1976

D. R. Uhlmann

Amorphous Ferromagnets

74-49 SPAR-IV June 1977

A. E. Lord and G. Wouch

Containerless Processing Technology

76-20 SPAR-IV June 1977

T. Wang